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Editor

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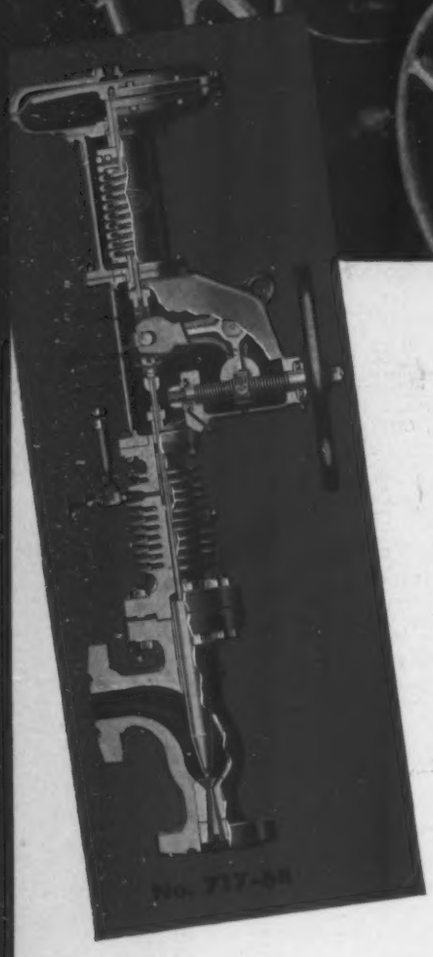

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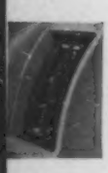
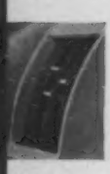
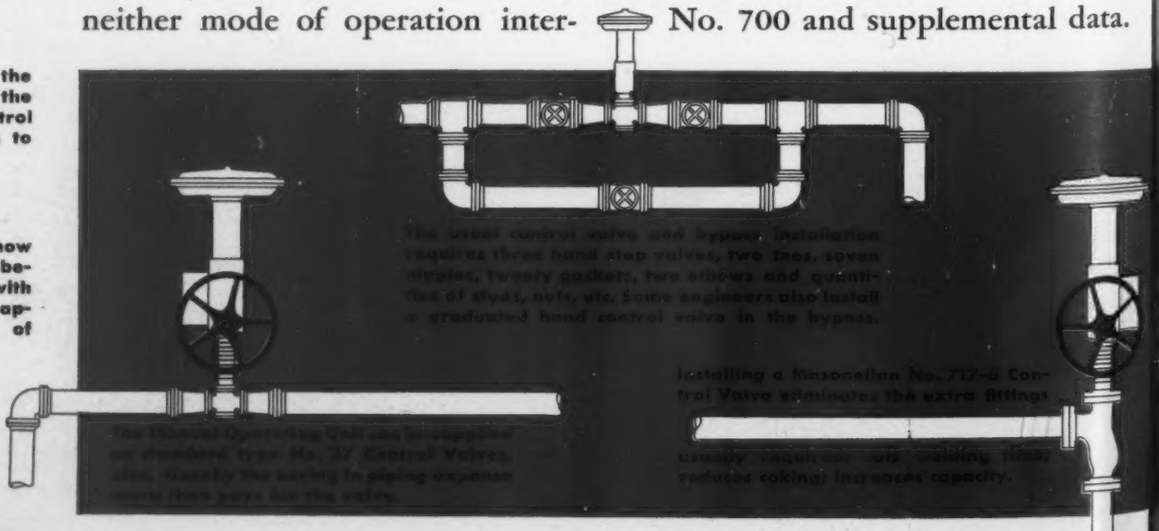


The indicators show the valve wide open and the stops set for 100% control range from full open to closed.



Here the indicators show a 25% control range between ½ and ¾ open with the valve positioned approximately midway of the range.



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Progress in Metallurgy *and its Application to* Petroleum Refining

ERNEST E. THUM
Editor, Metal Progress
Cleveland, Ohio

SEVERAL months ago, when the editor of THE REFINER AND NATURAL GASOLINE MANUFACTURER asked me to prepare an article "correlating latest developments in the metallurgical field as applied to petroleum refining" (those were his words), I started asking questions of such oil men as I met from time to time, and found that their principal problem connected with metal was to keep it from corroding away or burning up. There seemed to be a striking unanimity in their replies. Corrosion by condenser water, more or less brackish, and by hot petroleum products with or without sulphur and chlorine compounds, and oxidation by furnace gases while under heavy stress seemed to be the destructive causes working on tubes, pressure vessels, condensers and valves.

This statement may seem trite in this place, but it is a necessary preliminary to the metallurgist's feeling that great progress has been made in the last 15 years in the improvement of the common steels and the discovery and commercial production of special alloys to withstand these attacks. As a matter of fact, refinery equipment of plain carbon steel or carbon-molybdenum steel is handling most of the crude petroleum year in and year out; for corrosive crudes from certain fields the refiner goes first to 5 percent chromium-molybdenum steel, then 9 percent chromium steel, and finally to 18 percent chromium, 8 percent nickel stainless. New catalytic processes for cracking the heavy fractions and polymerizing the gases (except in one instance) will not increase the severity of service, now at about 700° F. and 1000 pounds per square inch.

On the other hand, condenser tubes give the refinery man a good deal of trouble when the cooling water is briney and the crude is corrosive. Each case is a problem in itself, and sometimes ferrous and non-ferrous alloys within the price range for the job have not been suitable. Admiralty brass is used in most condensers, but combinations of free chlorides, ammonia and H_2S will ruin it in short order; recourse may then be had to various silicon bronzes, aluminum brasses, 70-30 copper-nickel, and even duplex metals wherein the inner lining resists the vapors and the outer shell resists the brackish water.

It boils down to this: In almost all instances there is now available a metal or alloy for equipment which will outlast the probable life of the process—

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what with the rapid evolution of the refinery operations—but it is frequently at a price that makes the refiner hesitate. For severe service good engineering design for new vessels would indicate a composite structure, made of modern heat-resistant steels for strength and containing a corrosion-resistant metal for a protective lining, but such designs very often cost too much—more than the old plan of using the cheap, plain carbon or low alloy steels with from ¼- to ½-inch extra metal for a corrosion allowance, protecting them with gunite cement which is readily applied and easily repaired, and intending to replace the vessels at fairly frequent intervals.

COST OF ALLOY STEELS

Price of special high alloy steels is something that may warrant discussion. The purchaser may figure the pounds of iron, chomium and nickel in a ton of high alloy at their current unit quotations and arrive at a figure far less than he is quoted for the finished alloy in plate form. A little reflection will show that this is not a correct way to get a true picture of the situation. In fact, the refinery engineer can put himself in the steel maker's place, thus: Gasoline is the principal product of his refinery, the reason for his industry's existence; every mechanical and chemical device is centered on its production in huge quantities, and that's the reason it can be sold to the ultimate consumer at 12 or 13 cents a gallon (plus the inevitable taxes). The automobile driver may wonder why lubricating oil (a "by-product") costs 25 to 35 cents a quart, not thinking of the mass of special research behind the product, the cost of devices for a sharp separation of fractions far out of proportion to the volume of material collected, and the special plant for refining and dewaxing this oil to the last degree. Gasoline corresponds to the plain carbon steel of the metal industry; 90 percent of our facilities are engaged in the production of millions of tons annually of ordinary steel sheet, plate, bars, strip, wire, pipe, structural shapes and rails, and we are able to make it at 2½ cents a pound. Alloy steel (and especially high-alloy steel) is a specialty like lubricants; their diversity is so great that large orders and the economies of mass production are missing; specifications are so strict that inspection costs are high and there is an inordinately large proportion of scrap, useless except for remelting at further large losses; suitable raw materials are costly and special control is required at all steps; unusual and expensive surface finishes are the rule; large costs of preliminary development and of engineering service to purchasers must be absorbed. When all these things are reviewed, the base price of 18 percent chromium steel sheets of 32½ cents a pound does not seem so far out of line. This is 13 times the cost of plain steel; compare it with lubricating oil that costs ten times as much as gasoline. If further evidence is needed, consider the financial statements available at this writing of three leading companies specializing in high alloy steels—Crucible Steel Company of America, Allegheny Ludlum Steel Corporation, and Rustless Iron & Steel Corporation. Their consolidated balance sheets for 1937 and 1938 show 3-1/3 million dollars net income before dividends, which is certainly a meager amount to earn on sales of $150,000,000.

Returning from this digression into the distasteful but inevitable consideration of costs, it may be repeated that a variety of corrosion and heat-resistant metals—even steels—are now available, and the problem of the refinery engineer is to find the one (or the combination) which will give him the most economical life, the least cost per hour's operation when all the elements of the problem are included. The steels rely on an increasing amount of chromium in the alloy to resist attack by oxidizing liquids or gases and as is doubtless well known, the corrosion resistance and the scaling resistance increase more or less proportionately with the chromium content. That is to say, for the most severe conditions the highest chromium alloy will be required. Silicon and aluminum may be added to intensify the anti-scaling effect. At something less than 20 percent chromium, however, mechanical and fabricating difficulties appear in the iron-chromium alloys, principally related to the fact that they are "non-hardenable irons" rather than "steels," and as such cannot be heat treated to a fine-grained, tough condition. (In metallurgical terminology: Low carbon, 18 percent and higher chromium-irons are ferritic alloys, and coarse-grained ferrite of this sort is quite brittle.)

Fortunately, a relatively small amount of nickel will convert these high chromium-irons into hardenable steels. For instance, 2 percent nickel added with 18 to 20 percent chromium to the iron with the inevitable carbon will produce an alloy which may be hardened and toughened quite effectively by the traditional heat treatment of quenching and drawing. It is a true "steel," and the structural changes involved in the hardening process include a thoroughgoing recrystallization, which enables the metallurgist to produce fine-grained, tough material, much improved in strength. (The metallurgist would imply all this by saying that this is a "martensitic" alloy.) The additional nickel does not harm the corrosion resistance—quite the opposite. The steel therefore has been very popular in England, especially in places where it is exposed to sea water or other brines, or salty air, and where it is in contact with copper alloys (like bronzes) and electrolysis might be feared. It is available in this country, as well as modifications ranging down to 1 percent nickel in 16 percent chromium, but these varieties have not been able to make much headway against the higher nickel alloys now to be mentioned.

AUSTENITIC ALLOY

A considerable amount of nickel, say 8 percent or more, converts an 18 percent chromium ferritic alloy into an "austenitic" alloy. By this the metallurgist means that the microstructure is again of a single phase, but the crystalline structure (arrangement of atoms in space) is quite different from that in ferrite. Even though the atomic arrangement is so stable that recrystallization and hardening by heat treatment is impossible, austenite is essentially more ductile than ferrite at warm to frigid temperature, and these austenitic alloys (such as 18 percent chromium, 8 percent nickel steel, or "18-8" for short) are therefore quite tough and readily fabricated, and their strength may be increased greatly by cold work. In fact, the corrosion resistance of the chromium is so enhanced by the nickel that this alloy type is the most popular in America, despite its high price. The principal difficulty is that it might be regarded as a "universal alloy," and some sad mis-applications would follow.

Many minor variations in the analysis of the above

alloy types have been used by the petroleum industry in tubes for cracking stills—and I think it is fair to say, with surprisingly satisfactory results. The principal metallurgical difficulty has been to supply metal which remains stable during thousands of hours at temperatures up to 700° F. Even the microstructure of our old reliable plain carbon steel is unstable under these conditions. That is to say, the minutely dispersed iron carbide crystals which harden and strengthen the iron itself (the ferrite matrix) tend to dissociate, and the resulting particles of soft graphite are no longer a strengthener. However, this change does not **always** occur; for instance, R. W. Moore, metallurgist for Socony-Vacuum Technical Service Laboratories recently showed me the microstructure of a plain carbon steel tube withdrawn after 70,000 hours (ten years) of service because the wall thickness had been reduced by corrosion or erosion, and while the steel was considerably softer than originally, it was still amply strong and there was no trace of graphitization. He is of the opinion that vigorous discussion of interesting and perplexing changes found in the microstructure of still tubes withdrawn from service has caused an unwarranted worry over the adequacy of present-day metals. Of course, such discussions are of value to the steel makers, helping us interpret our products in terms of the customers' needs, but in reality, refinery troubles due to metal are rare; more failures are caused by operating errors, accidents, abuses or drastic conditions imposed on the metal beyond the designer's control.

At any rate, it is not surprising that these relatively new chromium alloys would have to be modified as experience accumulated in specific applications. An instance is the 4 to 6 percent chromium steel tubes first installed, I think, about 12 years ago by E. S. Dixon in the Port Arthur Refinery of The Texas Company. These operated excellently, but a number of brittle breaks, showing steel of no ductility, occurred during shutdowns when the tubes were being cleaned of coke with mechanical knockers. Apparently this steel remained tough and ductile while hot, but some unknown change occurred which caused them to become brittle after cooling to winter temperatures, only to recover their toughness again at operating temperatures. Whatever the nature of this change, it has been prevented by adding .5 percent of molybdenum to the alloy, and the straight chromium steel is now practically obsolete. An interesting variation is the addition of titanium to the improved analysis. (Titanium, by the way, forms such a stable carbide that it locks up almost all of the carbon in the steel so tightly it cannot enter any of the hardening reactions—in fact, it converts the steel into something like a softer iron of little hardenability.) These titanium-bearing tubes have been used in cracking stills to a small extent, but have found their largest application in air preheater tubes, and later in high-pressure superheater tubes. There are at least 20 superheaters in large central stations which contain the 4 to 6 percent chromium, .5 percent molybdenum, titanium analysis. This material is of definite advantage on account of its greater oxidation resistance; easy weldability, and high ductility. It is much easier to fabricate; since it is an "iron," complicated heat treatments are not necessary after hot forming or welding, as are required on the 4 to 6 percent chromium steels which do not contain titanium.

Pursuing this subject further, the 16 percent chromium-irons, as has been noted, are susceptible to grain growth and brittleness. This is quite marked in the regions of welds, and has somewhat restricted their use in the chemical industry, where the alloy is otherwise almost ideal for nitric acid manufacture. It has been discovered, however, that a stronger, tougher alloy is produced by introducing nitrogen, which acts as a grain refiner and perhaps also as a grain-growth inhibitor. At any rate, such modifications in the alloy, the use of 18-8 stainless steel welding rod or electrodes, and proper heat treatment of the finished job have corrected the trouble.

Finally, some peculiar actions of the 18 percent chromium, 8 percent nickel steel have caused intensive research, some of it of a pioneering nature, for these "austenitic" steels are so new that we have no background of experience. Most of the troubles are due to the fact that the austenitic microstructure, wherein the iron, nickel, chromium and carbon are mutually dissolved in a solid solution, is a metastable structure and given time at a moderate heat (in the range 800 to 1500° F.) a little carbide forms and separates out in small particles. If this occurred throughout the crystals indiscriminately, we would have essentially the same thing that occurs when plain carbon steel is hardened by quenching, but unfortunately the separation occurs for some unknown reason almost exclusively at the grain boundaries. This would not be fatal, except for the fact that the carbides are very high in chromium and deplete the surrounding film of metal of this corrosion resisting element; hence any corrosive agent more readily penetrates the alloy along the grain boundaries. Failure under these conditions is rapid and complete. The cure is to keep the carbon as low as consistent with good steel-melting practice and then fix it as a carbide insoluble even in the austenite. For this the element columbium is best adapted, and 18-8 steels stabilized with columbium are widely used for welding and for service above 700 or 800° F.

Another trouble with the pitting type of corrosion in 18-8 has not yet been completely solved. It had best be described and borne in mind, so disappointment may be avoided. About five years ago stainless steel gasoline tanks and fire lines were installed in some American naval vessels, but they rapidly failed by pit corrosion. The corroding solution was stagnant sea water, or more or less polluted harbor water, incapable of damaging the metal by general corrosion. Since many ship parts of stainless steel in contact with moving or aerated sea water were behaving excellently, the first guess was that the trouble was due to lack of enough oxygen or air in the solution to correct any damage done to the impervious oxide layer which is supposedly responsible for 18-8's corrosion resistance. That guess as to cause is as good as any, in the present state of our knowledge, even after a large amount of intensive research. Ferric chloride formed early in the exposure seems to be the bad actor in sea water corrosion; sodium hypochlorite and reducing rather than oxidizing conditions also cause rapid localized attack. Test methods have been devised which assess the susceptibility of individual samples, and by this means has been demonstrated the great importance of

metallurgical cleanliness in the alloy and perfection of surface finish. Best results are had if the smooth surfaces are pickled, polished and passivated by dipping in nitric acid. Likewise 2 to 4 percent molybdenum will prevent pitting, even on surfaces receiving no special preparation (1 percent copper for part of the molybdenum is also efficacious). For instance, an 18 percent chromium, 12 percent nickel stainless with 3 percent molybdenum will not pit under acidulated 10 percent $FeCl_3$ in six months, a solution sufficient to damage plain 18-8 in 4 hours. For grading such alloys 20-hour exposure to an acidulated 30 percent solution is necessary.

This pitting problem seems to be practically licked without the benefit of a scientific explanation!

STRESS CORROSION

As if only to give reason for metallurgists' existence, another disease dubbed "stress corrosion" has been most exasperatingly sporadic. (I am sure it is not unique to 18-8; it seems to be concentrated in that metal for that metal is frequently working under severe conditions—conditions wholly beyond the capacity of most other alloys.) Under certain undefined conditions of combined stress, temperature and corrosion the metal develops tiny surface cracks, like alligator skin, which destroy the toughness and progress inward to complete disintegration. The corrosion medium need not be very active—superheated steam and even tap water has been sometimes responsible. Probably most refinery engineers are acquainted with one or two instances. An example of unexplained variation in commercial stainless steel is the following:

Several tons of it were bought to line a number of vessels; the chemical analysis was chosen after tests showing it to be immune to the liquid being processed. At the very end of the job, a shortage developed and enough more sheet to line two or three nozzles was supplied from stock. In service the nozzle linings crumbled quickly; all the rest of the sheet has been perfectly satisfactory. The two lots of 18-8 are indistinguishable chemically, physically, and metallographically. The metal in the linings has been found to resist phosphoric acid, and the nozzle material does not—and this is now an added acceptance test for 18-8 in that particular service—quite pragmatic, for there is no phosphoric acid in the liquids being handled.

I hope that by this time the reader may not conclude that there are so many "bugs" in high alloys he had better stay away from them. If he can, he is lucky in having simple conditions to contend with in his refinery. If crudes or fractions are more corrosive, he can remember Mr. Moore's statement, already quoted, that "discussion of occasional failures has caused an unwarranted worry over the adequacy of present-day metals." He can take as an example Standard Oil Company of California, which has thousands of 18-8 tubes in cracking still service. Their first cost is high, but the oldest ones are now ten years old and apparently as good as new. Their life seems to be indefinite. That does not mean that there is never a failure. Replacements have been necessary due to intergranular cracking and oxidation leading inward from the fire side. "Stress corrosion" is the commonest explanation, although K. V. King, materials engineer, is not convinced that any hypothesis so far advanced will fit all the facts and explain why, now and then, one tube develops surface cracks whereas all its neighbors are unscathed.

The statement is frequently made that large pressure vessels cannot be made of solid stainless steels because of the cost. Without implying that this is untrue, I would like to express the thought that frequently it will be found that a bi-metal construction is fundamentally the best for this reason: Chemical stability is a surface phenomenon, whereas high static strength or creep resistance is a deep-seated phenomenon, and it is almost too much to expect one steel to combine chemical stability, high strength resistance to creep at high temperatures—all three properties, each one of which taken singly is not easy to produce, even in a specialized alloy. To make matters worse, when the three elements chromium, silicon and aluminum are present in sufficient amounts to give chemical stability at the surface, they derange the normal relationships between the metal and its strengthening carbides; the alloy is a nonhardenable iron, as explained already. What is more natural, then, that for high pressure service a composite should be formed with a thin lining having highest resistance to gas penetration and chemical attack and a thick-walled outer shell to take the pressure, made of ductile steel with high creep resistance. These items of equipment must, of course, be kept under close observation and, in the most severe services, replaced frequently.

Such situations have already been met in the chemical industry, where the limits of commercial metals have already been passed. Practical limits in the converters and reaction chambers used in the synthesis of ammonia and the hydrogenation of coal are now on the order of 1000° F. and 1000 atmospheres (15,000 psi. pressure). Under these conditions hydrogen actively reacts with carbides in steel and oxides in copper; recourse may be had to linings of oxygen-free silicon bronze, carbon steel stabilized by tungsten, and austenitic stainless steels.

There are also some new processes in the petroleum research laboratories which will go beyond the normal limits of 700° F. and 1000 pounds per square inch pressure. For example, a metal tube is needed to withstand say 1600° F. and 50 pounds per square inch pressure for long periods of time; in addition to the high temperature requirement, the metal must not oxidize or carburize. Rather a tough assignment! Probably higher temperatures would be used if a cheap metal could be obtained to withstand such conditions. Expensive high alloys are available to withstand about 5000 pounds per square inch internal pressure on small diameter tubes and 900 to 1100° F., but undoubtedly a lower priced alloy would be very welcome. At the present time an 18-8 stabilized with titanium or columbium, with about 2 percent silicon for its anti-scaling affect, and tungsten or molybdenum to enhance creep resistance, would be the metallurgist's probable recommendation.

This brings up the matter of "creep" or resistance to very slow plastic flow at elevated temperatures. As is doubtless well known, this phenomenon has been painstakingly investigated. An enormous amount of the data has been published in 1938 by a Joint Research Committee of the American Society of Mechanical Engineers and the American Society for Testing Materials in a "Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys." Briefly the method of test consists of

heating a specimen under tension in a protective atmosphere, observing length changes with the passage of time and plotting a time-elongation curve for the given steady load at the given steady temperature. If the metal is then beyond the limit of its capacity the curve will have four characteristic portions: (a) Elastic stretch which occurs almost instantly, (b) the succeeding plastic flow which rapidly settles down to a low figure, (c) the portion, longer or shorter in time, wherein the extension is at this very low but steady rate, and (d) approach to fracture, when the rate of stretch is steadily increasing. If the metal is at its theoretical "limiting creep stress," portion (d) will be missing, (c) the extension at a low but steady rate, will continue indefinitely. Loads below that limit will cause portion (b) to merge into (c) at a lower and lower rate—the creep theoretically reaching zero eventually.

It can be seen that such testing takes a very long time, and the final results will change with temperature and with stress, so a systematic statement for very few steels is available. While there may be no such thing in reality as a "limiting creep stress," H. W. Gillett in a recent lecture before the American Institute of Mining and Metallurgical Engineers told of one sample of 0.35 percent carbon steel having been under test for 20,000 hours and still stretching very slowly but uniformly. Steel, therefore, **can** be stable (in this sense) at high temperature. Those coarse-grained steels that have been tested have been stiffer at high temperatures than fine-grained ones of similar analyses. Likewise well-deoxidized "quality" steels perform much better than the "rimmed" type used for sheet and common bar stock.

As to composition and constitution, the carbon should apparently be kept in a medium range, alloyed with molybdenum or tungsten, and the microstructure contain its carbide as thin platelets ("lamellar pearlite") rather than in finely dispersed particles ("sorbite"). Users of high pressure, high temperature equipment should also remember that elevated temperatures increase the tendency for the micro constituents in steel to move toward structural equilibrium—that is, for the carbides to agglomerate in larger rounded particles, and this "spheroid structure" has the least ability to resist creep. Likewise the tendency of carbon to revert to graphite, as already mentioned, is another possibility.

Perhaps the ultimate steel for high temperature strength will be one properly alloyed and heat treated so it contains in supersaturated solution a substance that at operating temperature will very slowly precipitate a cloud of submicroscopic particles throughout the grains, thus continually strengthening them. In other words, the metallurgist may be able to utilize the almost universal phenomenon of "precipitation hardening" that is the basis of the art of hardening aluminum and copper. He would adjust the composition of the metal for the proper strengthening rate at the proper temperature, without developing undue brittleness under shock.

In fact, such alloys are already known. Take only one described by Francis B. Foley of The Midvale Company in *Metal Progress* in May, 1936. It had the following composition: Carbon very low, 0.03 percent; manganese, 0.16 percent; silicon, trace; nickel, 5.03 percent; aluminum, 2.59 percent. Forgings of this alloy had a Brinell hardness number of 310, corresponding to a tensile strength of about 150,000 pounds per square inch. Immediately after quenching the alloy was very soft, but on holding at 1000° F. the hardness reached and remained at 365 (tensile strength 180,000 pounds per square inch). Long stay at higher temperatures caused what the metallurgist calls "over-aging"—that is, the hardness and strength gradually slid off the peak. All this is typical of the common phenomenon of "precipitation hardening," and in this case the action occurs without appreciable carbon in the steel.

Many other alloys of this sort were found by W. P. Sykes when studying the iron-cobalt-tungsten alloys in the development of a new tool material. One carbon-free material analyzing 70 percent iron and 30 percent tungsten will age to its maximum hardness and stay there at 1300° F., retaining this hardness and strength indefinitely at any lower temperature. This hardness is C-54 on the Rockwell scale, and corresponds to a tensile strength on the order of 275,000 pounds per square inch (measured at room temperature). This alloy is cited merely to show what can be furnished the petroleum industry when it is prepared to pay $3 or $4 a pound for its materials.

Of course, creep resistant steels are available at very much less cost. The petroleum industry has had available and has utilized those inexpensive steels containing 0.20 percent carbon and 0.75 percent molybdenum, primarily developed for the super-power steam plants. Likewise some special alloys of moderate cost have been developed by Timken Steel and Tube Company which have interest to the petroleum industry. From experiments at the somewhat critical level of 1000° F. it is known that creep can be retarded by alloying elements such at molybdenum, tungsten, vanadium, manganese and chromium. The greatest gain in creep strength usually results from a comparatively small but definite addition of each alloying element. When the maximum economical benefit has been derived from the addition of a single element, further increase of creep strength can be obtained by the introduction of other alloying elements. In this manner excellent resistance to creep was developed in electric furnace steels of low total alloy content, as shown by the following table:

CREEP STRENGTH OF LOW ALLOY STEELS AT 1000° F.

Composition	1 Percent Creep in 100,000 Hrs. (Lbs. per Sq. In.)
0.15 C	2,700
0.15 C, 0.50 Mo	10,700
0.15 C, 0.75 Si, 1.25 Cr, 0.50 Mo	15,000
0.15 C, 0.75 Si, 2.50 Cr, 0.50 Mo	5,500
0.10 C, 0.40 Si, 5.00 Cr, 0.50 Mo	7,000

In these steels manganese does not contribute to corrosion or oxidation resistance and is of minor importance in building up creep strength at 1000° F. Silicon, chromium and molybdenum, on the other hand, are clearly elements which can be added to steels in various combinations to produce comparatively inexpensive alloys capable of satisfying many service requirements in oil refineries. The principal function of silicon in such compositions would be to furnish oxidation resistance, chromium the corrosion resistance, and molybdenum the toughness and creep strength. The 1¼ chromium steel is usually interesting. It has exceptionally high creep strength in the temperature range 1000 to 1200° F. Two to three times the corrosion and oxidation resistance of carbon steel is being obtained with this alloy, based on three years' service in refiner-

ies. The steel does not air harden, nor become "temper brittle," and is useful in parts requiring high strength where corrosion and oxidation are not unduly severe. It costs about half as much as 4 to 6 percent chromium steel (which is also an electric furnace product).

The above may be cited as a "low alloy, high strength" steel developed especially for high temperature service. Its mention will probably remind the reader of the multitude of such steels recently put on the market by various steel companies, to be used as rolled, without heat treatment, for structures or machine parts requiring more strength, atmospheric corrosion resistance or abrasion resistance than the common carbon steel, and yet be weldable, easily fabricated, and of low cost. Doubtless there are many places where such steels for lightweight construction would be economical in refinery service, but a vast literature is already available about them.

Enough now has been said to indicate that the metal industry can supply the petroleum refiner with some steels that will resist corrosion at high temperatures and other that will resist stress. It has also been indicated that it is frequently best to combine these two; make a strong vessel of cheaper steel and line it with the more noble alloy. There are several ways of making these so-called "bi-metals." One is to place two plates of corrosion resistant metal face to face and seal the edges all around by welding. A second similar pair is placed face down on the first, welded all around the edges and the composite slab rolled down, and edges sheared so that the finished plate separates into two clad pieces. This method has been especially successful in the plant of Lukens Steel Company for the manufacture of steel clad with nickel or the high nickel alloy (inconel). Another is to start with a slab of stainless steel facing with carbon steel backing, and a thin sheet of carbon-free iron between, heat in a reducing atmosphere, and weld all three together by rolling at a proper temperature. Still another is to plate the pieces with electrolytic iron on faying surfaces before heating for rolling and forging.

Modern welding methods are utilized by the principal fabricators of vessels for the chemical and petroleum industry. For instance, M. W. Kellogg Company starts with a thick plate of correct carbon steel and covers one surface with overlapping heavy beads of high alloy weld-metal, using automatic machinery and electrodes properly selected so the deposit has the correct chemical composition for the intended service. This slab is then heated and rolled in a plate mill to proper thickness and surface smoothness. (The United States Steel Corporation has patented a similar process—although I believe it is not yet in production—where the welded-on layer is made by a carbon arc traversing systematically and melting a mixture of crushed metals or ferro-alloys, thus forming the alloy layer from its raw materials.) A. O. Smith Corporation utilizes this system: A steel plate of proper thickness with carefully cleaned surface has a sheet of corrosion-resistant metal placed on top and the two joined by a multitude of spot welds on say ¾-inch centers, automatically made by electrical resistance, the current being brought in through hard copper wheels of large diameter which roll over the surface. The Babcock & Wilcox Company produces the bond between corrosion resistant alloy and steel backing by resistance roll welding covering the entire surface.

In any event, these flat bi-metal plates are then formed, pressed or spun into shapes which when assembled form the vessel under construction. Automatic electric welding is utilized for the joints; usually the steel backing would be welded first, and the joints in the lining sealed next, using a technique, inspection and test to insure that there is no dilution of the alloy in the inner joints and no pinhole leaks through the lining.

Many such vessels, some of a size and weight that reach the limits of transportation equipment, are giving good service. In each case the metal lining must be carefully chosen by preliminary tests adequate to indicate the type of alloy required to resist the corrosion, whether it should be stress relieved to avoid failure by stress-corrosion, or whether it should be quenched from high temperature or stabilized to prevent intergranular attack alongside the welds. Neglect of these precautions has been responsible for some unsatisfactory linings placed in existing steel cylinders by refinery staffs.

Before closing the matter of pressure vessels, it might not be amiss to say something about the necessary data for design. "Creep of metals" is getting to be a familiar term, but there is still some doubt how best to use the data which are being accumulated. The attitude taken by a designer of turbines, for instance, wherein no more than one or two thousandths of an inch per inch distortion is permissible even after 20 years' service, will be entirely different from that of a boiler designer, in whose product ten times as much creep in half the time would be of small moment. Since most creep data are reported on the basis of 1000 hours (6 weeks), comparatively few tests have extended to 5000 hours (7 months), and the record length is in the neighborhood of 2½ years, the propriety of extrapolating these results to expected life of 10 to 20 years should be seriously questioned by the designer of high speed machinery.

Fortunately for the designer of pressure vessels, a rougher approximation is quite useful. As pointed out by T. McLean Jasper in *Metal Progress* for February, 1939, the designer of a pressure vessel to operate at high temperature needs to consider the "long-time ultimate strength" and "long-time yield strength" of his material for the working conditions of temperature and pressure (including occasional over-pressures) and the effects of corrosion. "Long-time strength" is found by immersing several specimens of a particular steel in a lead bath (to avoid surface oxidation) at a definite temperature, loading each to a different constant stress, and measuring the time until failure occurs—or until the load is maintained without failure for a very long time. The time for failure versus the test stress for each specimen is then plotted and the resulting curve drawn which applies, of course, for a single test temperature. It is debatable whether, for instance, a 0.25 percent carbon steel piece which did not fail after 2000 hours at a stress of 18,000 pounds per square inch **would** have failed if the time had been doubled, but even on the supposition advanced by some that the stress versus time plots as a straight line on log-log paper, the rupture strength for a given steel at a given temperature and for a given time may be estimated with sufficient accuracy for successful design. In Mr. Jasper's practice, a lower factor of safety, based on this long-time ultimate strength, would be used than though the equipment were to operate at ordinary temperatures, for the reason that short overloads are resisted by ductile steels by a much higher factor of safety at high temperatures than though it were in service at ordinary ones.

A somewhat more difficult situation exists in the manufacture of valves. Valves are very critical; the

refinery engineer is not to be criticized for leaning over backward for safety in this detail, and even then installing them in duplicate to be doubly sure. The maximum demands of the steam power plant are readily met with carbon-molybdenum and other low alloy steel castings, but austenitic chromium-nickel castings are necessary to meet the worst conditions existing in oil refineries. J. J. Kanter of The Crane Company tells me that one petroleum company desires valves to operate at 1350° F. and 3000 pounds per square inch, but he could not recommend going higher than 1200° F. and 2000 pounds per square inch. Such valves would be an austenitic 18 percent chromium, 8 percent nickel steel, stabilized with silicon against the intergranular precipitation of carbide which occurs more readily in castings than in wrought products. Corrosion, cavitation and galling or seizure on the seats of such valves are avoided by welding layers of stellite (an alloy of cobalt, chromium and tungsten) and grinding to shape. Erosion by impingement of hot fluids is largely avoided by good design that "streamlines the flow." Turbulence causing cavitation is probably more often the source of trouble than direct impingement of a high velocity stream.

Flange bolts represent a very serious problem at temperatures of 1000° F. and above, for they are originally set up under high tension, and any creep will cause a slight relaxation with consequent leakage. Some 11 varieties are listed in tentative specifications A 193—37 T adopted by the American Society for Testing Materials in 1937. Of these the best of the low alloys is probably the one containing 0.40 percent carbon, 1 percent chromium, 0.35 percent molybdenum and 0.25 percent vanadium. After a special "normalizing" heat treatment giving a relatively coarse grain the relaxation will be hardly one quarter that of the chromium-molybdenum steel very popular for this same service. Safe temperatures are now at least 150° F. higher than 10 years ago, but the improvement is due only in part to improved steels; much has resulted from better design of the flanges, improved metallic gaskets, and design of threads. Users also realize that wide temperature fluctuations will surely cause trouble with bolts.

This section should not be closed without some mention of the castings required for return bends in cracking stills and tube hangers. Usually the return bends are of castings to match the analysis of the tubes. Troubles here are generally due to variations in foundry practice, and sometimes these can be minimized by a change in the design. Even so, the refiner is well advised to shop around for the most competent foundryman rather than the lowest price. Speaking of conditions known to exist for mechanisms in heat treating furnaces, operating under load up to 1500 or even 1700° F., a notable improvement in serviceability in these more severe conditions, estimated as at least 50 percent by one user who disclaims being an enthusiast, has been achieved in the last two or three years by closer attention in the foundry to raw materials and analysis, melting and deoxidation practice, pouring temperatures, molding methods, sand control and X-ray inspection. For services where rapid changes in temperature are encountered, or where large temperature differentials in single pieces exist, nothing but alloys of the high nickel type will stand up for any reasonable length of time. These are indicated in the following table:

Code designation	NC-4	NC-2	NC-1
Nominal analysis			
Nickel	35%	60%	65%
Chromium	15%	12%	18%
Iron	balance	balance	balance
Suggested design strengths			
at 1200° F.....	7000 psi.	4400 psi.	
at 1400° F.....	3300	2100	
at 1600° F.....	1500	800	
at 1800° F.....	550	200	
at 2000° F.....	200	100	

The compositions are varied to include molybdenum, aluminum and titanium to improve high temperature strength and refine the grain.

A great many words have now been said about metals and services which are reasonably well fitted to each other. Something ought now be included about a problem that is really acute in many locations—that is, the rapid corrosion of condenser and heat-exchanger tubes. In a plain heat exchanger with oil on both sides, plain carbon steel is usually satisfactory; in fact, it is frequently found that laboratory corrosion tests made to simulate plant tests do not give as good results as actual service has proven.

Condenser tubing is another thing. It is a matter of record, for instance, that in one large Texas refinery there are about 230,000 such tubes of all sorts and their average life is three years. Admiralty brass and red brass are used very largely. Their life is various; successive bundles installed in the same condenser shell that is giving trouble may last one year, two years, or six months, with no correlation as yet to operating variables (by design or accident), or the nature of the cooling water as affected by rainfall or other climatic conditions. Other types of tubing are continually under test, but their life is also so various under ordinary operating conditions that the staff at this refinery is not convinced that any of them is sure to last enough longer to warrant their extra cost.

Frequently something that is done to protect one part of the equipment causes much trouble in another. For instance, magnesium chloride in some crudes forms hydrochloric acid which is deleterious to the iron towers and shells. To counteract this corrosion of the iron, ammonia is sometimes added at some point in the operation. If this ammonia is not carefully controlled and a further combination of hydrogen sulphide results, this condition produces a very rapid corrosion in the copper alloys.

Admiralty metal, due to its relatively low copper content, relatively high zinc content and the presence of 1 percent tin, is intrinsically a good material to resist corrosion from the oil side whether due to sulphur, or sulphur combined with slight acidity. Also this same alloy has been found to be basically sound for resisting most corrosion from saline waters. The main difficulty has been that, at temperatures found in oil refinery practice, it is quite susceptible to dezincification on the water side.

The copper metallurgist, familiar with the fact that red brass, that is, alloys containing 85 percent of copper and higher, have been entirely immune to dezincification in domestic plumbing service, would suggest that as a logical substitute. In many installations they have lasted 1½ to 3 times as long, at no increase in cost. Other reports are of even more rapid

[*Continued on page 226*]

Application of Stainless and Heat Resistant *Steels* in Refineries

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FOR a period of years the petroleum refining industry and the steel producers have cooperated whole heartedly in the development of special materials of construction for the processing of petroleum products. When the refinery engineers desired a metal of certain properties to meet definite requirements, the metallurgists of the steel producers tackled the problem and endeavored to fill the need. Conversely, when the steel industry developed a new alloy, the refineries were among the first to determine where in their processing the new material could be economically applied. This excellent cooperation has resulted in widespread application of the corrosion- and heat-resisting alloys to refinery equipment to the mutual benefit of both producers and users.

It has been estimated that the annual toll for corrosion in refinery equipment amounts to millions of dollars. To curtail this as much as possible, the refinery engineers, well known for their progressiveness, have adopted stainless and so-called semi-stainless alloys to a wide variety of applications. Undoubtedly the best known of these applications are those where the tonnage is also the greatest. Seamless tubes containing 2 percent and upwards of chromium are widely used for still tubes, vapor lines, hot oil transfer lines and heat exchanger tubes. For application where ordinary steel tubes would have short life due to high temperature corrosion by the petroleum products or due to oxidation, the use of the chromium alloys has proven very economical.

The stainless alloys are today the accepted materials for refinery valve trim. Here the martensitic alloys (capable of being hardened by heat treatment) are generally used. However, recently, there has been a trend toward the use of the austenitic chromium nickel alloys, 18-8, for trim for valves for the handling of the cold (temperature of 400° F. or lower as a rule) refinery products, due to the better resistance of these alloys to the reducing acids occasionally present in the products when they are at temperatures below the dew point of water. For the handling of products from the catalytic polymerization units some of the valve trim has been of the 18-8 Moly, Type 316 alloy. This material, which contains 2 to 4 percent of molybdenum in addition to the usual 18 percent of chromium and 8 percent of nickel, is per-

* Presented before the Refining Division,, Oil World Exposition, Houston, Texas.

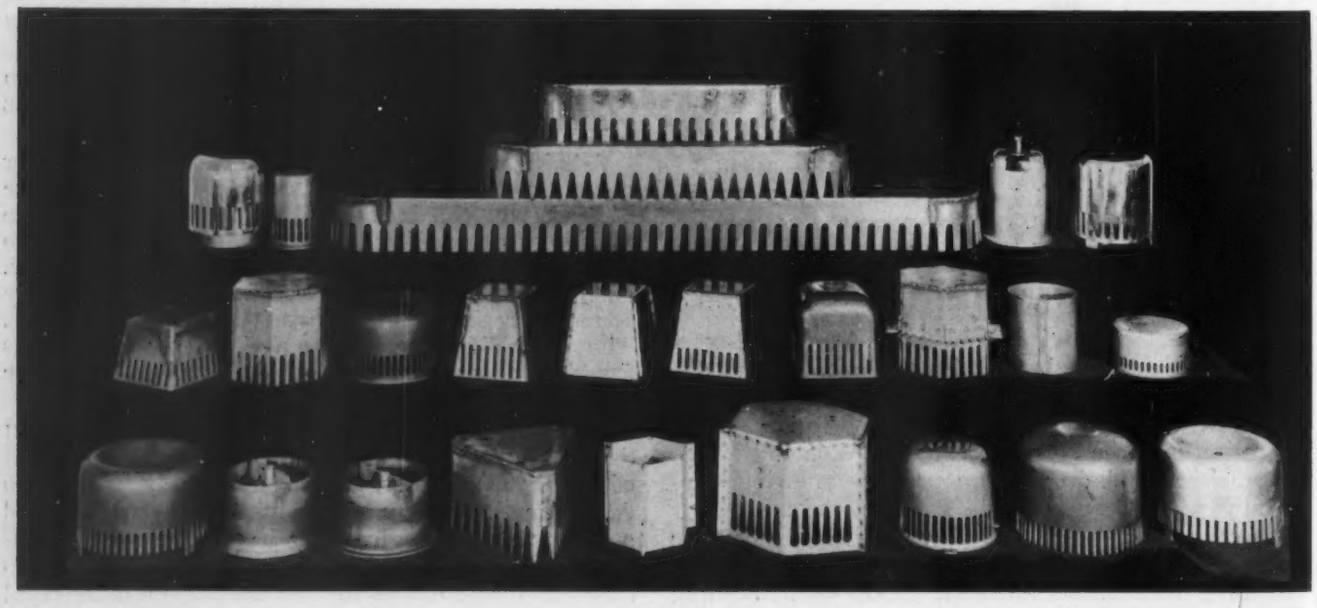

FIGURE 1

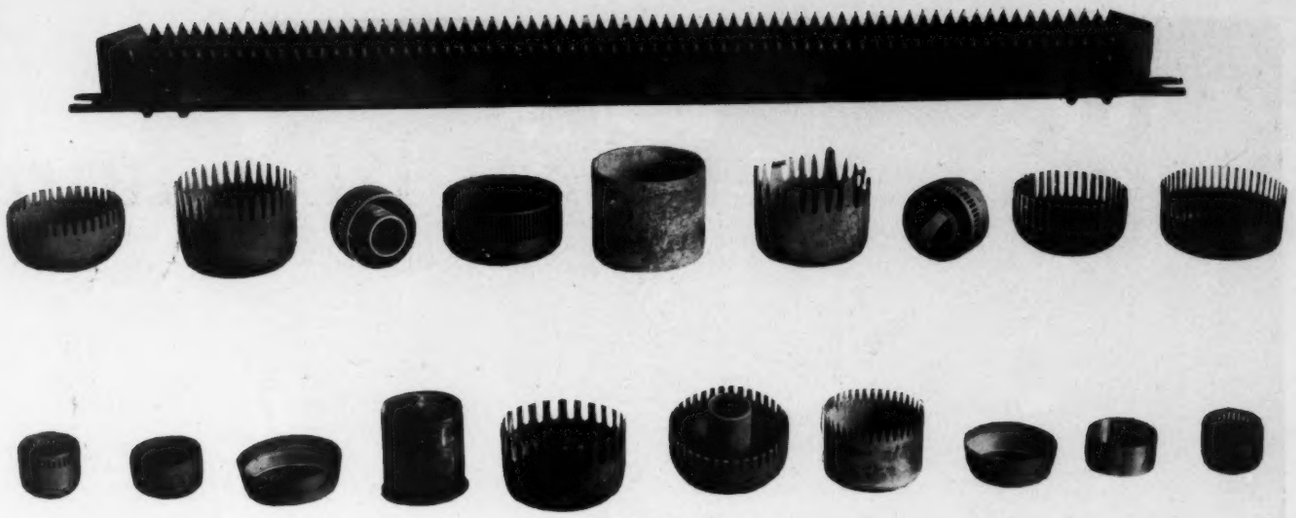

FIGURE 2

haps the most resistant, of the so-called stainless steels, to wet corrosion when the acids present are reducing in character. It is believed that as the corrosion resisting properties of this 18-8 Moly alloy becomes better known to refinery engineers, its use will become more widespread.

PUMPS

Pumps of various kinds are now liberally fitted with stainless steel. The pump rods are commonly of the austenitic 18-8 alloy, whereas the pump shafts of centrifugal pumps are generally made from the martensitic straight chromium alloys. Plungers for hot oil pumps have been successful when made from 4-6 percent chromium molybdenum seamless tubing, also when made from 18-8 tubing. Valves and valve ports of the plunger type and the piston type of hot oil pumps are frequently made of the stainless alloys. In some cases the pump body itself has been made of these alloys. In the case of the centrifugal hot oil pump, stainless steel shafts, impellers and liners for the bodies are often used when handling corrosive crudes.

Other generally adopted applications of the stainless steels include liners for nozzles of towers; gaskets; orifice plates; thermo-couple wells; baffles; supports and spacers, as well as the tubes of heat exchangers; tray support angles for bubble towers; floats for control valves, and furnace dampers. They have also been very successfully used for high temperature air preheaters on stills as well as flue gas recirculation systems. It is believed that the application of the heat-resistant steels for high temperature air preheaters will increase materially once refinery engineers appreciate the marked improvement that has been made in this type of equipment during the last few years.

Stainless steels have been proved economical for the prevention of corrosion in systems used for the removal of hydrogen sulfide and mercaptans from refinery gases and vapors. These steels are beginning to be applied for such uses as guy wires on large torches or flares; sheave wheels for tank float gauges; cables for swing lines; small size pipes for control and instrument lines; discs and seats for vapor valves on tanks.

The stainless steel bubble cap is no stranger. It has been used in fairly large volumes; both in natural gas plants handling sour gases, in the absorbers and stabilizers in the refineries and in the refinery bubble towers. Its principal advantages, in addition, of course, to its outstanding corrosion resistance, is the lack of breakage of the teeth or slots and its extreme lightness. The lack of breakage of the teeth likely will have a particular appeal to those refineries desiring sharp fractionation. Another advantage to be obtained, if desired, is a greater number of teeth or slots per cap, thus allowing more intimate mixture of the vapor and the liquor on the tray. This is due, of course, to the thinness of the material and to the fact that the slots or teeth can be easily punched. The thin cap also allows for more useable area on the tray, since there is less metal in the wall of the cap to take up dead area.

Stainless steel bubble caps .0625-inch thick have been used in bubble towers handling cracked West Texas crude for over 8 years with no apparent evidence of corrosion. They have been made in a wide variety of designs as evidenced in Figues 1, 2 and 3.

Among the more recent applications of stainless steel to the refineries has been the extremely light weight, relatively inexpensive, complete bubble tray for the hot end of the system. In the past, while it was generally acknowledged that the best way to combat corrosion by hot petroleum products was to

FIGURE 3

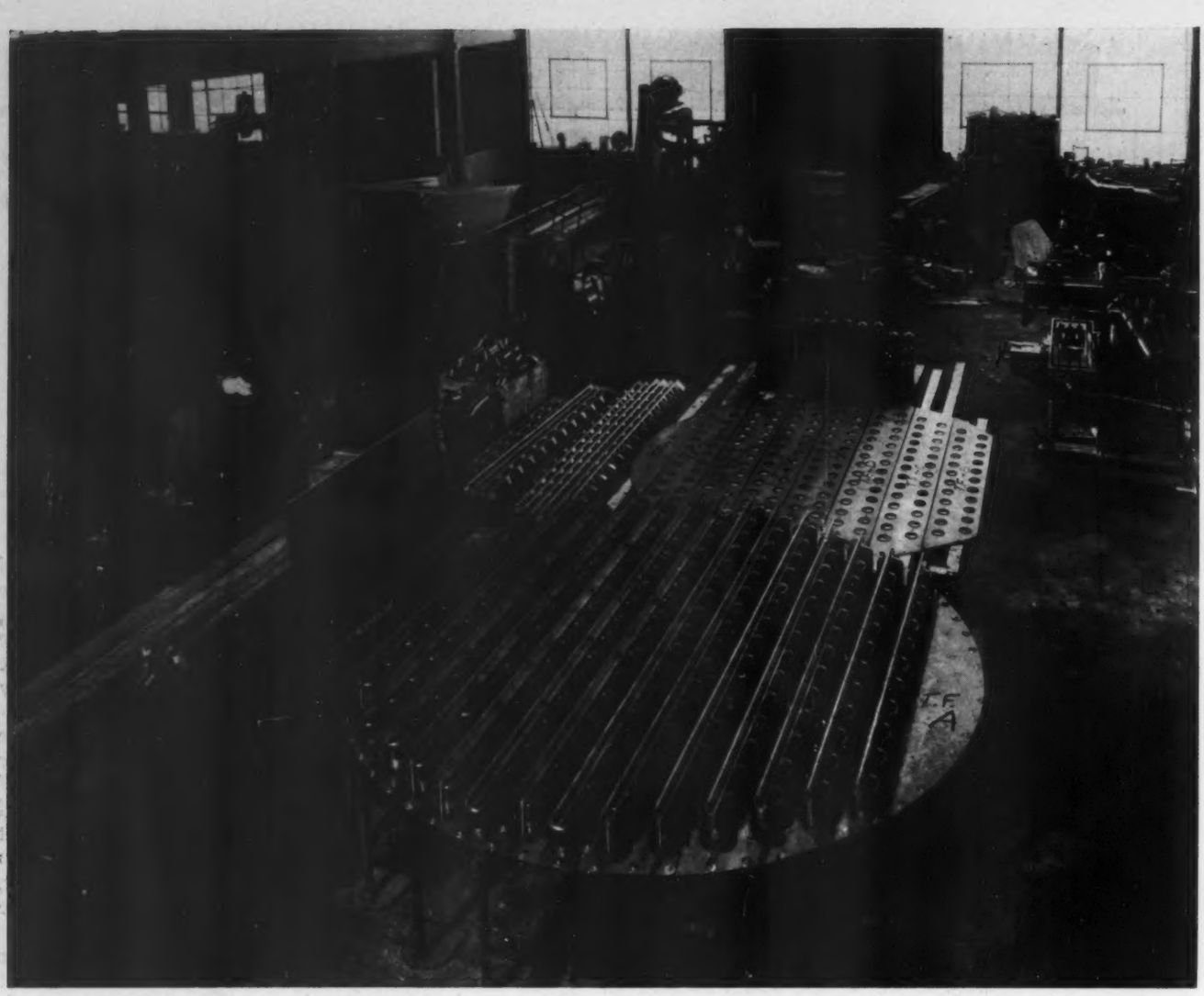

FIGURE 4

use stainless steel, the cost of items such as bubble trays, down-comers, rain decks, baffles and weirs of stainless steel, when made in accordance with conventional design, was such as to exclude their use except in those cases where a severely corrosive crude was being handled. However, by applying the experience of the aircraft and the rail car builders in light weight design, the cost is brought down to what is believed to be a fairly competitive figure. The stainless alloys having 11 percent or more of chromium appear to be particularly immune to corrosion in the temperature range 1200° F. to 400° F. in petroleum refining processing which makes the use of thin, relatively inexpensive sections possible inasmuch as little, if any, corrosion allowance need be added. Necessary stiffness and strength can be obtained using thickness no greater than .0625-inch by the application of skill in design, engineering and fabricating. The principle advantage of the stainless trays is, of course, the extensive life due to the corrosion resistance. Breakage or spoilage should be at a minimum due to the tough and ductile properties of the material. Coking and labor for decoking will be appreciably less due to the fact that the smooth, dense surface does not allow the coke to get a "toe hold." The extreme lightness makes for ease of installing and removing the trays. The lack of hazard to the workman when handling the light tray sections should appeal particularly to the safety men. A bubble tray for an 11-foot tower complete with bubble caps, risers, hold down bars, down-comers and weir fabricated of .0625-inch thick 17 percent Cr. and supporting trusses fabricated of .078-inch thick 17 percent Cr. as illustrated in Figure 4 weighs only approximately 800 pounds. The tray complete with supporting trusses but not including the caps, risers or hold down bars weigh approximately only 300 pounds. It will support a 4-inch liquor level with a maximum deflection of $\frac{1}{8}$-inch. The rupturing load of the individual supporting trusses was designed with a factor of safety of five. This tray can be made appreciably stiffer, if desired, by adding only slightly to both the weight and cost by merely deepening the supporting trusses shown in Figure 5.

Figures 6 and 7 show another design of bubble tray and the supporting trusses. Figure 8 shows still another design where the required rigidity is obtained in .0625-inch thick 17 percent Cr. by merely forming the sheet into channel sections. This tray does not require supports underneath. From these photographs it can be seen that the relatively inex-

pensive stainless steel tray can be fabricated in a wide variety of designs.

LINERS FOR REACTION CHAMBERS

Stainless steel liners for reaction chambers, flash towers, evaporators, bubble towers, vacuum towers and heat exchanger shells have been used for some time. There is no question but that properly applied, they fully protect the steel from the high-temperature corrosion and are almost wholly free from trouble. When building a new tower stainless steel liners can be satisfactorily applied by the fabricating shops by a variety of methods. Generally a low carbon 12 percent chromium stainless steel sheet or strip is used although 18-8 has also been successfully applied and used. The life of the liners appears to be practically unlimited, since the high chromium content apparently gives perfect resistance to corrosion and prevents any contact of the oils or vapors with the steel shell.

Recently there has been a rapid development in the application of the stainless steel liners to existing steel vessels. Towers lined in the field 9 or more years ago are still giving entirely satisfactory service. While it is not claimed that a field lined job can be done as cheaply nor as perfectly as a tower lined in a fabricating shop, nevertheless, it appears to be a well established fact that field lining can be done both satisfactorily and economically. It is estimated that in the last 2 years alone over 100 vessels have been field lined with stainless steel.

There are numerous methods used to apply the liners. The most common method consists of using .08 percent carbon maximum 11 percent to 13 percent chromium in strips of from 0.125 to 0.070-inch thick. The width of the strip is generally limited to that which can be conveniently taken in through a manway, but it is generally no wider than 24 inches. The lengths are limited to 5 or 6 feet for ease in fitting. These strips are punched or drilled with holes $\frac{5}{8}$ to 1-inch diameter (the more experienced the welder is, the smaller the holes, and the thinner the material; however the $\frac{5}{8}$-inch diameter is considered to be the minimum) on various centers of from 3 to 5 inches (the higher the operating temperature of the vessel, the smaller the "center" of the holes). They are then rolled to fit the diameter of the tower. In the case of domes of towers, the stainless strip is generally first cut to make "orange peel" segments, then punched or drilled and finally shaped. Application consists of snugly fitting the liner against the shell, which must be thoroughly clean, and then welding the periphery of each of the holes to the steel shell, cleaning the weld of slag, sealing each hole, and finally fastening and sealing the edges of the individual sheets. Due care must be taken to assure that no imperfections in the welds exist to allow oil or vapors to get back between the shell and the liner, and that the liner is attached to the shell at sufficiently short intervals to withstand the stress occasioned by expansion and contraction when the vessel is coming up to temperature and when being taken off stream. For those who do not wish to undertake this lining work themselves, there are

FIGURE 5

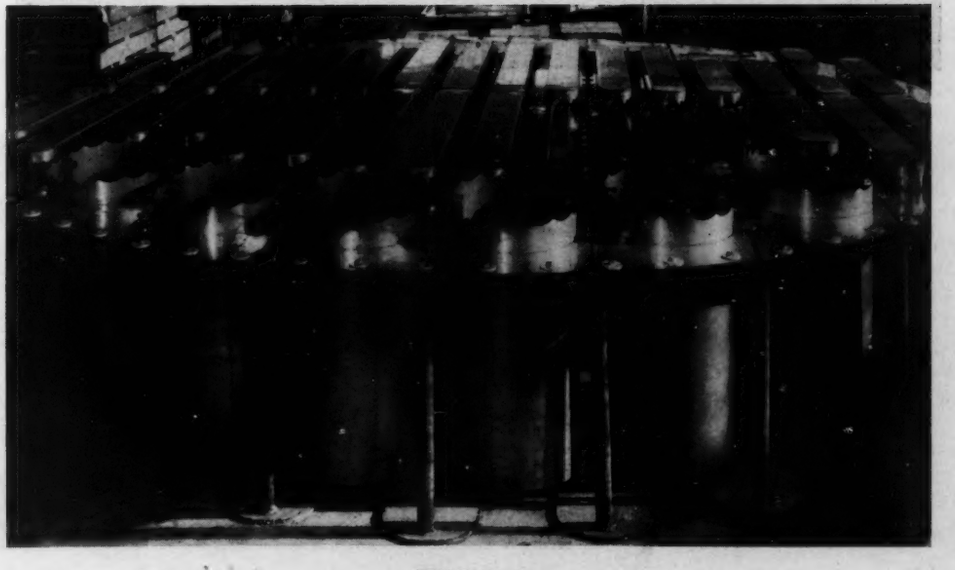

FIGURE 6

FIGURE 7

available contracting firms who are equipped to do the work on a turn key basis.

PATCHING

Another application of stainless that is of particular interest is the use of 18-8 welding rod for the salvage of worn steel parts and for the protection of new steel parts against both erosion and corrosion. Apparently most refiners are now doing some of this stainless weld overlay work, but it appears that there has been little or no exchange of information, since one refinery will be using the method for one application and completely neglecting numerous applications used by other refiners. Among the uses of the 18-8 weld overlay are the building up of eroded, corroded or "wire cut" bodies of control or flow valves; building up of the plungers of hot oil pumps; facing flanges; building up the bonnets of valves; lining of spools, vapor line ends, bends, junction boxes, headers and plugs; lining and building up pump bodies and valve parts on pumps; lining heat exchanger heads and tube sheets; building up of both the plugs and bodies of plug cocks; lining of check valve bodies; and lining inside the stainless trim on the gates of gate valves. The resistance to erosion and corrosion of these 18-8 weld overlay surfaces is truly remarkable. Since most of the weld overlay work requires finishing by machining, it is desirable to use a weld rod giving the most free machining weld bead. Unfortunately, the regular free machining 18-8, Type 303, does not lend itself well for use as weld rod and therefore 18-8 Ti., Type 321, containing roughly 0.5 percent titanium, appears to be the best suited. The titanium burns out when passing through the arc and the weld bead in consequence contains microscopic particles of titanium oxide and nitride which appear to act the same way to give free machining qualities to the material as do the sulfide inclusions in free machining Bessemer Screw Stock.

Further cooperative effort on the part of the petroleum refining engineers and the steel metallurgists is certain to bring forth developments of new or different applications of stainless steels. Successful applications are much more numerous at present than was the case only a few years ago. The future should see the stainless and heat-resisting steels growing in importance and usefulness in refineries to the end that marked reduction in the annual cost of corrosion of plant equipment will be achieved.

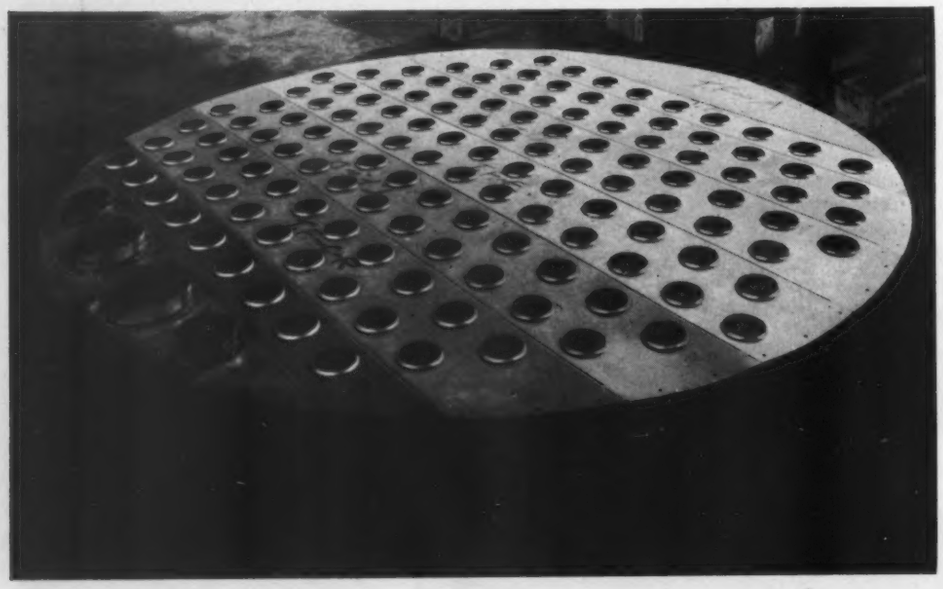

FIGURE 8

FIGURE 9

Gunite Liners *for* Pressure Vessels

E. S. DIXON
The Texas Company, Port Arthur, Texas

MANY vessels in refining units are protected against corrosion with non-metallic lines. Principal among these is "gunite"—"gunite" being the name applied to that concrete which is applied with a cement gun. Gunite is particularly adaptable for liners which are installed in the vapor space of soaking chambers if a service life of but one year is expected from the liner and a relatively inexpensive liner is desired. The liners described in the following discussion are, therefore, those from which only one year's wear is required. They are installed in the vapor space of soaking chambers wherein the temperatures are from 850 to 900° F., with corresponding pressures of from 350 to 400 pounds per square inch. Gunite has been used also for liners in fractionating towers, but for these it has not always been found successful.

In describing the liners the following points will be considered:

1. **General process of application.**
2. **Preparation of the vessels for the lining.**
3. **Installation of the reinforcing.**
4. **Installation of the gunite, including the materials used.**
5. **Curing of the gunite.**
6. **Mechanics required to install the gunite.**
7. **Costs.**

This paper was presented at Ninth Mid-Year Meeting, American Petroleum Institute, at the Roosevelt Hotel, New Orleans, La., May 17, 1939.

PREPARATION of the vessels to be gunited is very important, and must not be slighted. It is essential that all grease, coke, old liners, or dirt be removed completely from the walls prior to guniting. The surface must be clean—this being accomplished in old vessels first by scaling off all old deposits and then by sandblasting; in new vessels, sandblasting alone generally will suffice. After sandblasting, all the loose sand and dirt are blown out of the vessel with air to minimize the rusting of the shell. For blasting, ordinary building or river sand has been found satisfactory, and much cheaper than any special blasting grit. Air pressure for use of this cheaper sand must be maintained at from 80 to 100 pounds for successful operations.

Reinforcement for the gunite is installed immediately following the cleaning of the vessel. This consists of 3 x 3 electric-welded 10-gauge ungalvanized mesh supported on ¼-inch diameter vertical rods. These rods are crimped every 12 inches for the purpose of welding them to the vessel walls, and also to hold them away from the wall in order to facilitate the installation of the mesh thereon. The mesh is fastened to these vertical rods by tying in place, every 8 to 10 inches, with short wires such as the tie wires used in concrete work. Welding could be used for fastening the mesh in place, but it is more expensive as regards materials, and also requires skilled craftsmen. After installation of the reinforcement and just prior to the actual installation of the gunite, the vessel is washed out thoroughly with water to remove the last trace of any dirt.

APPLICATION

The application of the gunite is made with a special air-driven machine through which a continuous flow of material is possible. This machine takes a dry mixture of cement and aggregate, and transports it by means of air pressure through a suitable hose to a nozzle, where the necessary water is added to the mixture for the hydration of the cement. This gun or machine shoots the mixture onto the wall or form with such force that apparently 25 to 30 percent of the sand or aggregate is lost. This sand, in a true sense, has not been lost; it has served a very useful purpose—that of tamping the concrete or gunite compactly and tightly in place.

The gunite material, as ordinarily used, consists of 1 part by volume of Portland cement (other cements have been tried, but are found to produce no better gunite for our conditions than portland cement) to 2½ parts of sand. In plants in the Gulf Coast region, river sand is used which has an excess of fines, i.e., a large percentage of it will pass through a 30-mesh sieve. This sand produces an exceptionally dense and compact gunite, and discussions with various cement representatives lead us to regard this material as an important factor in the success of our gunite.

At this point it is well to re-emphasize the necessity for cleanliness in handling gunite work. Considerable dust arises during the shooting of the gunite, which must be kept blown off the bare surfaces as well as off the reinforcing. The dust, if allowed to remain, will prevent the necessary bond, and cause a failure. In applying the gunite, an area of approximately 2 feet square is built up, with a more or less circular motion of the nozzle, until it is the required thickness. The specification on thickness calls for the gunite to be 1 inch. This is a minimum thickness, the actual thickness varying from 1 to 1½ inches, as it is impractical to apply gunite to an exact thickness of 1 inch in such cramped quarters as a soaking chamber. It is very important that each section be built to full thickness before going on to the next section, as the dust in the vessel will settle upon it—and in a very short time, even in as short a period as 1 or 2 minutes, be in such condition that a cleavage

plane would be formed should other gunite be placed on top of the original. Layered work is not satisfactory in liners.

No finishing is required on the gunite after it has been applied, although oftentimes the surface is very lightly brushed off. This must be done with extreme care, as any pressure on the gunite towards the vessel wall would tend to draw the water from the gunite and break the bond between it and the vessel wall. Loss of bond between the liner and vessel walls means a failure very shortly after the liner has been placed in service.

CURING

Curing of gunite liners in a vessel is comparatively easy as compared with many concrete structures. In vessels, it is a simple matter to install a water spray in the top of the vessel and produce a fog-like condition. (A lawn sprinkler on a ¾-inch pipe entered through any convenient opening produces a very nice spray that is ideal for curing.) This fog-like condition or spray is maintained for at least 3 days after the gunite first has been allowed to set up for 24 hours. After this moisture cure, the liner is permitted to air-dry for 24 hours, and it is then ready for service. A steam cure, which is effected by closing up the vessel and introducing into it moist steam at a pressure of from 80 to 125 pounds per square inch—constantly bleeding out the condensate—produces, in 24 hours, the same results as are produced by keeping the gunite moist with water for from 3 to 5 days. In steam curing, as in the water curing, the gunite must be allowed to set up properly prior to the introduction of the steam, and it also must have the same air-drying period afterwards.

Application of gunite is an art gained only through experience. Men skilled in applying gunite in unrestricted areas or in the open are, as a general rule, unable successfully to shoot gunite liners in vessels without the training gained by actual experience. Men must apply several liners before they begin to learn the art, there being no set of instructions for the novice to follow which will guarantee a satisfactory liner. Practice alone produces results.

CREW

In this work a five-man crew generally is required, as follows: a nozzle man to shoot the gunite, a man to operate the machine, two men to prepare the dry mixture, and a supervisor. A minimum crew would require at least three men, as follows: a nozzle man, a machine operator, and a material man. But for the best results and for a speedy job, a crew of five is recommended.

COSTS

Any schedule of costs, of course, will apply only to the locality or localities in which the gunite liners are to be used; and will vary according to local labor conditions and prices for materials, as well as to the skill of the gunite crew. The following is a general average, which presumes that the job will be undertaken by a skilled contractor:

LABOR

	Per Square Foot
Removing old liner, or scale vessel	$0.20
Sandblasting vessel	0.04
Welding in the vertical rods	0.10
Tying mesh	0.04
Shooting gunite	0.15

MATERIAL

Mesh	0.05
¼-inch-diameter rods	0.03
Cement	0.04
Sand	0.01
Total cost	$0.66

It will be noted that the foregoing costs comprise only those items which apply directly to the liner. In addition, provision must be made for scaffolding for the workmen, transportation of the materials and equipment to the site, pipe work incident to the water and air supply, the cost of the water and the air, electricity and other incidentals—the cost varying for each of these items; in fact, it has been found to vary from $0.20 to $0.45 per square foot, thus making an over-all cost of from $0.86 to $1.11 per square foot of surface, on the basis of one year's service for the liner.

Progress in Metallurgy

(Continued from page 219)

failure of red brass in oils high in sulphur, wherein copper-lined admiralty, a bi-metal tube costing a 50 percent premium, has shown better service life, namely 1 to 2 times that of admiralty. Another possibility is the copper-nickel tubes (nickel 20 percent or even 30 percent) which have been so successful in high-duty condensers on steamships and warships.

At least two alloys have been specially developed by the brass industry, and are under extensive test, but their average life span is not yet determined. One starts with a copper-zinc alloy with copper low enough to prevent rapid attack by sulphur compounds but high enough to prevent dezincification, and adds aluminum for its known resistance to sulphur. The result is an alloy of approximately 82 percent copper, 2 percent aluminum, 1 percent tin, and 15 percent zinc—rather expensive because it is very difficult to pierce, draw, clean and finish.

D. K. Crampton of Chase Brass & Copper Company also informs me that admiralty metal tubes alloyed with antimony are giving very encouraging results. It has been known for some time that arsenic markedly decreases the susceptibility of all brasses to dezincification and therefore arsenical admiralty is a distinct improvement over normal admiralty. However, as is frequently the case, the correction of one difficulty leads to some other trouble. It was found, for instance, that the arsenical admiralty is susceptible to intercrystalline attack which sometimes causes premature failure. Recently it was discovered that antimony is fully as effective as arsenic in preventing dezincification, and the alloy so treated is entirely immune to intercrystalline attack. Therefore antimonial admiralty appears a more logical all-round material than either the base alloy or the arsenic alloy.

Midget Polymerization and Midget *Iso*octane Units

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Universal Oil Products Co., Chicago, Ill.

THERE are more than 50 UOP catalytic-polymerization units in commercial operation, design, or under course of construction at the present time. The capacities of these units, processing cracked or olefin-containing gases, range from 125,000 to 27,000,000 cubic feet; or, on a gasoline (81-octane) production basis, from 18 to more than 2,500 barrels daily. The combination of selective catalytic-polymerization and hydrogenation units produce from 50 barrels (midget size) to 800 barrels of *iso*octane fuel per day. The increased yield of gasoline ranges from approximately 2 to 8 percent, with an octane rise of 1 to 2 numbers on the refinery-gasoline output when processing naphtha, kerosene, gas oil, or topped crudes.

The midget catalytic-polymerization units particularly are adapted to the small refinery due to the low cost of installation and the increased yield and quality of the gasoline produced. The cost of units processing cracked gas at the rate of from 125,000 to 1,500,000 cubic feet (arbitrarily called "midget units"; daily gasoline production, 18 to 215 barrels) is approximately $15,000 to $35,000.

There is no cracking unit operating commercially today that is too small or too large for the installation of a catalytic polymerization unit to increase the volume and quality of the gasoline produced from a given cracking stock.

This paper was presented at Ninth Mid-Year Meeting, American Petroleum Institute, at the Roosevelt Hotel, New Orleans, May 18, 1939.

MIDGET catalytic poly units are in operation in many refineries. Five of these poly units have been selected for this study due to the differences in cracking stocks such as naphthas, gasoline, topped crude, combination of topping and cracking, and the variation of olefin content of the cracked gases produced which ranged from 19 to 41 percent. These poly units process daily from 125,000 cubic feet of cracked gas in the smallest unit to 800,000 cubic feet in the largest. The polymer gasoline produced by the smallest unit is approximately 18 barrels per day, whereas the largest unit yields about 100 barrels per day.

Midget poly units consist essentially of a heater, catalyst vessels, cooler, and a stabilizer. In order to minimize the size of the equipment required, and to obtain a long catalyst life, these units are operated at elevated pressures. As the charge to these small units is available either at separator pressure or stabilizer pressure, viz., at 50 to 200 pounds, a compressor is required to compress the gases to 500 pounds, in addition to the equipment outlined. The material used in the construction of these units is standard in every respect, with no special alloys necessary. The stabilizers used are of a packed-column variety rather than the bubble-deck type.

In the operation of these units, the poly-plant charge is picked up by the compressor and dis-

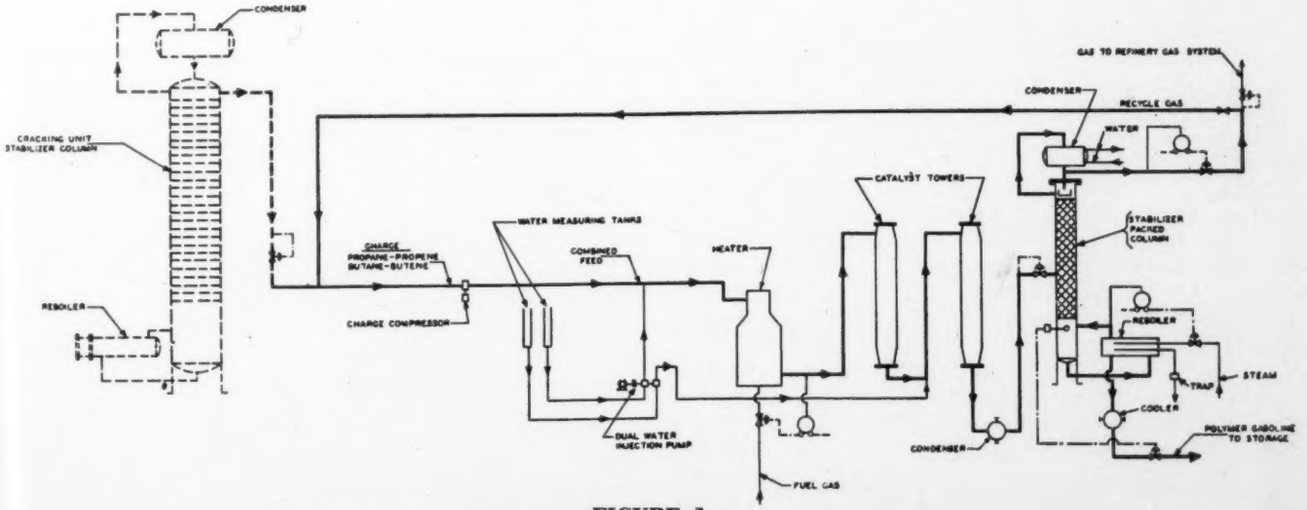


FIGURE 1
UOP Midget Catalytic Poly Unit

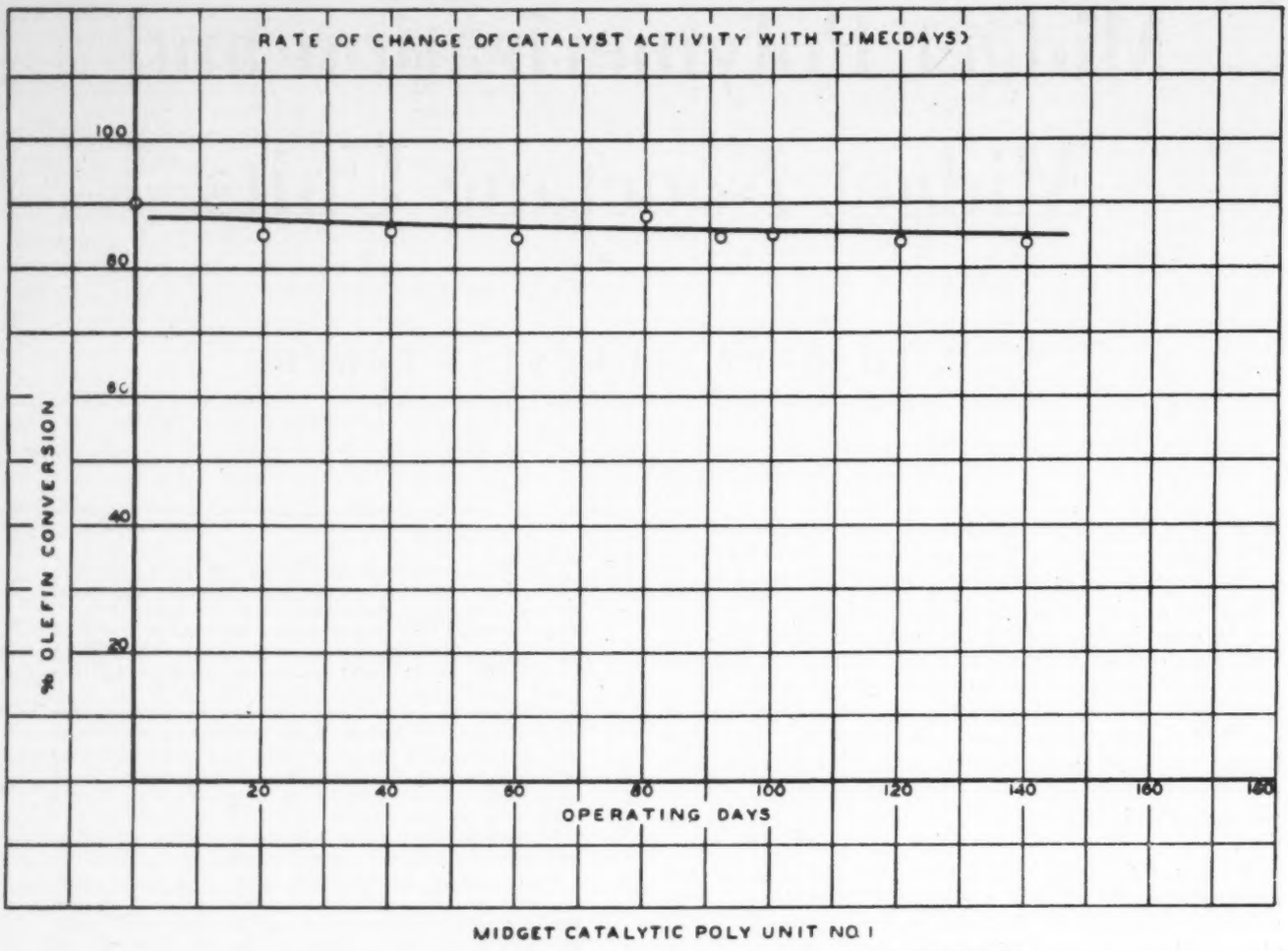


FIGURE 2
Rate of Change of Catalyst Activity with Time (Days) Midget Catalytic Poly Unit No. 1

charged to a heater where the temperature of the gas is raised to the order of 350 to 400 degrees F. The gas so heated then is passed in series through two tubes, usually made of 24-inch pipe, filled with solid phosphoric-acid catalyst upon whose surfaces the reaction takes place. The polymer product leaving the last catalyst vessel then is cooled to temperatures of the order of 165° F. and discharged directly into the stabilizer—where the polymer, together with the required amount of butanes to give the desired vapor pressure, is separated from the balance of the gas called "spent gas." A schematic outline of midget poly units is shown in Figure 1.

The poly units are constructed so that the operating conditions are maintained automatically and the amount of control necessary is similar to that required by a stabilizer or an absorber. The compressor is hooked into the gas system, and the gas from the stabilizer discharged in such a manner that a constant gas flow is maintained throughout the system. The hook-up is arranged so that the poly plant can be cut in or out at will without interrupting the cracking-plant stabilizing and gas-disposal system. Automatic controls are used to hold a constant inlet temperature to catalyst towers; a similar control is provided for the stabilizer reboiler. Pressures are regulated at the outlet of the catalyst towers and poly stabilizer. By proper manipulation of factors affecting catalyst-bed temperature, high olefin conversions and long catalyst life are obtained.

In some refineries where the olefin content of the cracked gas is high, a portion of the spent gas is recycled with the fresh feed in order to control the temperature rise which takes place in the catalyst vessels and which, uncontrolled, would yield a product having a high percentage of material boiling outside the gasoline range. The polymerization operation is completed on discharging the spent gas to the refinery fuel system and the gasoline to storage. The stabilizer gas has in general a B.t.u. content of approximately 1700 per cubic foot, which is reduced to 1300 after passage through the poly unit. The reduction in volume of the stabilizer gas is approximately 27 percent, which corresponds to approximately 6 gallons of polymer gasoline per 1000 cubic feet of cracked gas processed.

In general, it has been found that the most advantageous type of cracked gas to process is derived from the stabilizer. Two-thirds of the polymerizable hydrocarbons which are produced in cracking are included in this stabilizer gas stream. As the average quantity of stabilizer gas produced is generally one-third of the total gas produced in cracking, it is possible to produce two-thirds of the maximum yield of polymer in equipment one-third the size of that required for processing the total cracking-plant gas. However, from a process standpoint, it is entirely feasible to treat the total gas produced in cracking. The latter type of charging stock to a certain extent is advantageous; because its lower olefin content makes the matter of obtaining proper temperature control, on which catalyst life is somewhat depend-

TABLE 1
Cracked-Gas Analyses
(Charging Stocks to Midget Poly Units)

Midget Poly Units	No. 1	No. 2	No. 3	No. 4	No. 5
Composition		(Mol Percent)			
Hydrogen sulfide	0.15	1.8	0.33	1.0	0.35
Hydrogen	4.1	0.2	0.3	0.1	..
Methane	25.8	5.0	4.6	3.4	2.2
Ethylene	5.3	2.6	4.0	3.7	2.3
Ethane	16.9	18.9	20.3	16.4	10.7
Propene	15.0	17.1	17.9	23.4	17.8
Propane	23.3	44.1	29.5	27.8	25.1
*n*Butenes	2.8	2.8	7.4	9.8	16.4
*Iso*butene	1.4	3.0	7.4	5.0	6.8
Butanes	4.6	5.9	11.9	10.0	17.9
Propene-butenes	19.2	22.9	29.4	38.2	41.0
Polymer gasoline (gallons per 1,000 cu. ft. of gas)	3.7	4.4	6.1	7.6	9.5

ent, much simpler. There are poly units operating which process both types of gas as a single stream of compressed gas.

As the catalyst loses activity, the temperature rise due to the polymerization reaction decreases, as does also the average temperature of the catalyst. To correct this condition and to maintain the higher temperature, less recycle gas is used—which increases the olefin content of the combined feed; hence the temperature rise in the catalyst bed. By this manipulation of the recycle-gas volume and the inlet temperature to the catalyst reactors, the average catalyst-bed temperature can be adjusted to losses in catalyst activity—thereby increasing the catalyst life, and producing high-olefin conversion. A number of the midget plants in commercial operation still are operating after five months with the same catalyst charge. During five months of operation there has been little decrease in catalyst activity. One of the units has produced polymer in excess of 70 gallons per pound of catalyst. Figure 2 shows the life of catalyst and its activity over a five-month period, and gives indication that the catalyst will have at least double the present life.

The utilities required for a unit processing 500,000 cubic feet of gas are: fuel, 650,000 B.t.u. per hour; water, 4000 gallons per hour; process steam, 550 pounds per hour; compressor power, 35.

The operation of these units is simple. No additional labor is required, as the cracking-plant operators also control the midget poly units.

CRACKED GASES TO BE POLYMERIZED

Five midget poly units operating commercially were selected, having cracked gases containing from 19 to 41 percent propene-butenes to represent this type of operation. These units are called: No. 1, 2, 3, 4, and 5 for the sake of identification. The cracked-gas analyses for the five units are shown in Table 1.

The charge to midget poly plant No. 1 is a mixture of the pressure-still receiver gas and stabilizer gas. The hydrogen-sulfide content of these gases varies from 0.15 to 1.8 percent. When hydrogen sulfide is present in cracked gases under catalytic-polymerizing conditions, a reaction takes place—with the olefins forming mercaptans such as ethyl, propyl, and butyl.

When the sulfur content of the polymer gasoline is a factor to be considered, the gas should be freed of hydrogen sulfide before the polymerizing reaction. In connection with these midget units, simplified hydrogen-sulfide-removal units have been designed—although installations have not been made as yet. Their cost is in line with the installation costs of the polymerization units.

Midget poly unit No. 2, processing gas high in hydrogen sulfide, produced a gasoline containing 1.5 percent sulfur. In this case a means of treating was worked out which uses an absorber and the waste caustic from the refinery treating plant. When the sulfur content of the polymer gasoline is relatively high and waste caustic is not available, demercaptizing the polymer gasoline with strong caustic-soda solution, removing thereby 60 percent more or less of the sulfur present, may be cheaper than hydrogen-sulfide removal from the cracked gas. Polymer gasoline containing 0.3 or 0.4 percent sulfur is used, as such, or blended with the gasoline produced in the refinery to bring it down to market specifications. In some parts of the country 0.4-percent-sulfur-polymer gasoline is used and marketed with refinery gasoline containing a similar amount of sulfur.

SPENT GASES

The cracked gases after the olefins have been polymerized into gasoline leave the stabilizer of the poly unit with a low olefin content. The cracked gas, after being de-olefinized, is called "spent gas." Analyses of the spent gases leaving the stabilizers of the five midget poly units are shown in Table 2, the headings of which correspond to those of Table 1 of the initial cracked-gas charging stocks.

The propene-butenes in the spent gases range from 2 to 6 percent. The percentage of olefin conversion to polymer gasoline determined from the analyses in Table 2 and those given in Table 3 ranges between 85 and 95 percent. One plant averaged 95 percent of olefin conversion to polymer gasoline for a five-month period.

In order that a clear picture may be presented of the olefin conversion effected by the five poly units, it is necessary to consider not only the olefins in the spent gas, but also the butenes dissolved in the polymer gasoline. In the case of the poly units under study, the average vapor pressure of the gasoline produced is of the order of 25 pounds (18.8 to 30.9 pounds). The butanes-butenes present in the gasoline average about 30 percent of the high-vapor-pressure polymer. The olefin content of this C_4 fraction must be considered in determining the over-all olefin conversion. Table 3 shows the olefin content of the C_4 fraction (normal and *iso*butane and normal and *iso*butenes) included in the polymer gasoline.

In general, the butene content of the C_4 fraction in the polymer gasoline is consistent with the olefins in the outlet gas, although the differences are of

TABLE 2
Spent Gases

Midget Poly Units	No. 1	No. 2	No. 3	No. 4	No. 5
Composition		(Mol Percent)			
Hydrogen	5.4	0.4	0.2	0.2	0.3
Methane	31.6	10.3	8.5	5.5	5.6
Ethylene	6.1	2.8	4.0	3.7	3.5
Ethane	24.6	34.1	30.2	29.8	25.1
Propene	2.2	3.4	2.1	4.6	0.8
Propane	22.9	46.0	41.9	49.8	50.7
*n*Butanes	0.7	0.5	1.1	1.2	0.8
*Iso*butene	0.3	...	0.2	0.2	0.4
Butanes	6.2	2.5	10.2	5.0	11.6
Propene-butenes	3.2	3.9	3.4	6.0	2.0

TABLE 3

Midget Poly Units	No. 1	No. 3	No. 4	No. 5
	(Percent)			
*Iso*butene	1.7	1.5	2.5	1.4
Butene	12.3	8.5	23.9	6.1
Normal and *Iso*butane	86.0	90.0	73.6	92.5

wider range. It will be noted that the percentage of *iso*butene, as would be expected, is low in every case due to its ease of polymerization compared to normal butene.

There are usually three butenes present in cracked gases made up of butene-1, butene-2, and *iso*butene (2-methylpropene-1) varying in percentages—each having a different velocity of polymerization to gasoline under a given set of operating conditions. The *iso*butene polymerizes rapidly; butene-2 less so; and butene-1 with some difficulty.

From the data in Table 3 it is apparent that the results obtained on poly unit No. 4, at the time the samples were taken and analyses made, were inferior to those of the other four commercial units. However, an olefin conversion to about 90 percent polymer gasoline was possible by modifying the operating conditions.

Temperature affects the rate and degree of polymerization of olefin. A graphic representation of olefin conversion as a function of temperature change is shown in Figure 3. The data are shown in Table 4.

The analytical data presented in Tables 1, 2, 3, and 4 permits one to calculate the olefin balances, as a result of poly-unit performances.

The percentage polymerization of the individual olefin hydrocarbons present in the cracked gases is shown in Table 5.

In poly units No. 4 and 5 the percentages of ethylene in the cracked gases are, respectively, 3.7 and 2.3; propene, 23.4 and 17.8; *n*butenes, 9.8 and 16.4, and *iso*butene, 5.0 and 6.8. Ethylene is relatively difficult to polymerize compared to propene and butenes under the conditions of commercial operation of the poly units. All of the olefins in the cracked gases could be polymerized, but the product would boil largely outside the gasoline range. The data show that propene polymerizes readily—in both cases exceeding the average olefin conversion, and exhibiting appreciably increased tendency towards polymerizing, when compared with normal butene. In the case of unit No. 4, the failure of normal butene to polymerize is chiefly responsible for the relatively-low olefin conversion.

POLYMER GASOLINE

Polymer-gasoline yields based upon the cracked-

TABLE 4

Effect of Catalyst-Bed Temperature on Olefin Conversion

Average Bed Temperature (Deg. F.)	Olefin Conversion (Percent)
406	76.5
415	82.0
421	80.5
423	84.4
428	88.3
429	90.0
445	92.8
452	95.0

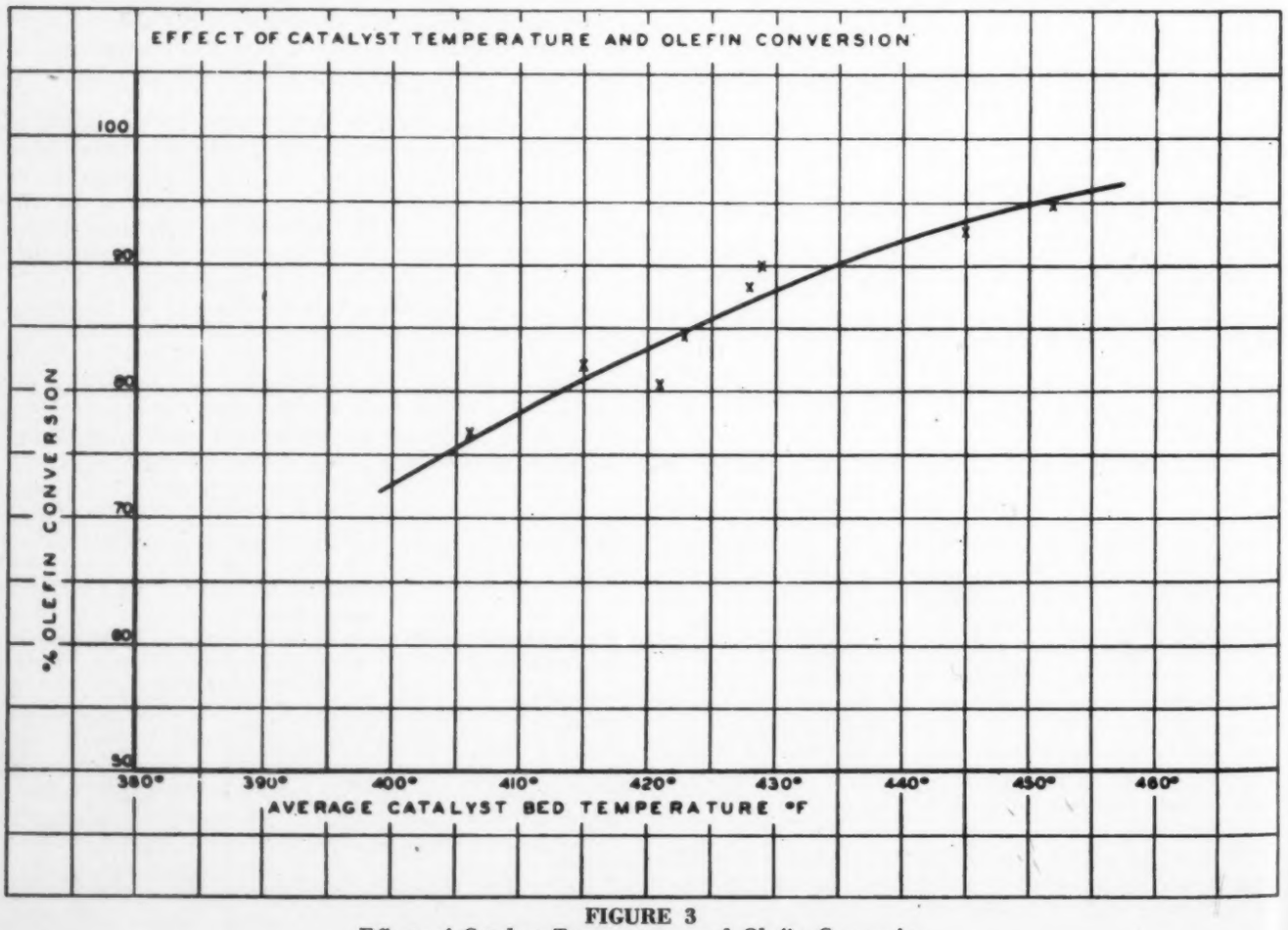


FIGURE 3

Effect of Catalyst Temperature and Olefin Conversion

TABLE 5
Percent Conversion of Individual Olefin Hydrocarbons to Polymer Gasoline

Midget Poly Units	No. 4 (Percent)	No. 5 (Percent)
Olefin:		
Ethylene	44	35
Propene	89	98
Butene-1 and -2	71	92
*Iso*butene	97.3	94
Average conversion (propene-butenes)	85	94.9

gas charging stocks to the midget units are dependent upon the percentage of propene and butenes present. The olefin content of the cracked charge to the five units under discussion ranges between 19 and 41 percent olefins. The yield of polymer gasoline, based upon a 10-pound Reid vapor pressure, ranges between 4.3 to 9.4 gallons per 1000 cubic feet of gas charged to the units. These volumes of polymer gasoline show an olefin conversion ranging between 85 and 95 percent. The higher conversion yield is for the cracked gas containing 41 percent olefin. Polymer-gasoline and olefin conversion rates are the average of more than five months' continuous operating periods for midget units using the same catalyst. The gallonage of polymer gasoline represented by this period of operation is between 38 and 70 gallons per pound of catalyst.

In the five polymer units under discussion the catalyst at the end of five months had a lower activity than at the beginning of the operation. The order of polymerization activity of the catalyst decreased when using a 41-percent olefin gas; from 98 percent conversion to 90 percent at the end of five months. When processing a 20-percent olefin gas, the catalyst activity decreased from 90 percent conversion to 80 percent—giving an average conversion of about 85 percent for a five months' operating period.

The analyses of the polymer gasoline for the five plants are shown in Table 6, and correspond to the gas analyses given in Table 1.

The analyses in Table 6 show that the Reid vapor pressure of the raw polymer product ranged between 18.8 to 30.9 pounds. It is possible and generally advantageous to operate the poly-plant stabilizer not only as a stabilization unit, but also as a means of effecting further butane recovery from the cracking-plant gases. It is possible to make available for polymerization all of the butenes which enter the cracking-plant stabilizer by deep stabilization. As a result, an increased yield of polymer gasoline is produced from the butenes normally contained in the 10-pound-vapor-pressure product. The recovery of additional butane from the gas discharged from the cracking-plant stabilizer and poly feed is necessary to make up the deficiency in vapor pressure and gasoline volume brought about by polymerizing the butene content of the C_4 fraction contained in the cracked gasoline. The increased yield of polymer gasoline as a resultant of deep stabilization is from 40 to 50 percent of that obtained when cracked gasoline with a Reid vapor pressure of 10 pounds is produced at the stabilizer. Deep stabilizing involves producing a 5- to 6-pound product, which generally requires maximum retention of butane in the poly-plant stabilizer, and gives a polymer of high vapor pressure.

TABLE 6
Raw Polymer

Midget Poly Units	No. 1	No. 2	No. 3	No. 4	No. 5
Reid vapor pressure, lb.	20.5	24.1	25	18.8	30.9
Gravity, deg. A.P.I.	83.0	73.0	74.0	71.0	77.5
ASTM Distillation in °F.:					
Initial boiling point	72	69	..	70	55
5-percent point	90	106	..	90	80
10-percent point	105	123	..	100	96
20-percent point	122	152	..	119	138
30-percent point	145	191	..	151	197
40-percent point	196	206	..	188	226
50-percent point	230	218	..	211	241
60-percent point	243	237	..	228	302
70-percent point	265	267	..	247	364
80-percent point	298	310	..	285	...
90-percent point	...	...	..	341	450
End point	415	370	..	408	450
Recovery, percent	89.5	87.5	..	94.5	75.5
Residue, percent	1.5	1.5	..	1.3	1.5
Loss, percent	9.0	11.0	..	4.2	23.0

The directly-produced gasolines of high vapor pressure by the poly units were stabilized to 10 pounds Reid vapor pressure. The quality of the products is shown in Table 7.

Inspection of the data for the polymer gasoline shows appreciable differences in the end point of the polymer. To a lesser extent, similar differences exist in the ASTM gum content of the various polymer gasolines. In the past we have considered that the end point of the polymer gasoline was essentially a function of the activity of the catalyst. With a fresh catalyst, high end points inevitably would result, but would decrease with loss in catalyst activity. The end point of the polymer then was considered to be essentially a function of olefin conversion. The operation of these units has shown that a considerable amount of control can be exercised over the end point and gum content of the polymer gasoline; and, at substantially constant olefin conversions, it is possible to vary the end point over a wide range. Fortunately, the operating conditions which are conducive to the formation of polymer gasolines of low end points, low ASTM gum and, to a lesser extent, good color, are likewise conducive to the maintenance of maximum catalyst life and activity.

SWEETENING OF POLYMER GASOLINE

In the five illustrated midget poly units, the sweetening operation (by either the UOP copper method or sodium plumbite) is carried out on the polymer product as produced prior to blending in three cases;

TABLE 7
Polymer Gasoline with a Reid Vapor Pressure of 10 Lb.

Midget Poly Units	No. 1	No. 2	No. 3	No. 4	No. 5
Gravity, deg. API	65.0	66.2	59.7	66.0	62
Color	*	*	*	*	*
Sulfur, percent	0.43	0.08	0.34	1.7	0.26
Mercaptan sulfur, percent	0.19	0.03	0.15	0.80	0.15
Gum:					
Copper-dish, mg. per 100 ml.	40	33	30	45	35
Air-jet, mg. per 100 ml.	10	..	1	11	10
Octane No. (L-3 method)	82	84.5	83.5	82.5	84
Blending octane	86	..	...	..	...
(in 70-octane gasoline blends)	90	..	94.5	88	97.5
ASTM Distillation in °F.:					
Initial boiling point	90	90	87	90	95
10 percent point	135	130	133	126	132
20-percent point	172	165	175	157	165
30-percent point	196	191	209	186	190
40-percent point	209	206	234	206	208
50-percent point	221	218	251	220	226
60-percent point	241	237	281	233	241
70-percent point	264	267	306	254	265
80-percent point	299	310	333	286	302
90-percent point	415	370	402	416	450
End point	...	...	...	..	...
Recovery, percent	98.5	87.5	...	94.5	75.5

* Light yellow.

TABLE 8

Polymer Sweetening and Blending Tests

Gasoline	UOP Catalytic Polymer Gasoline			Cracked Gasoline	Polymer—Cracked-Gasoline Blends
Sample No.	1	2	3	4	5
Treatment	Original Caustic, Washed Sour, Polymer Gasoline	Laboratory Doctor-sweetened Polymer Gasoline	Laboratory UOP Copper-sweetened Polymer Gasoline	Plant sweetened-Cracked Gasoline from Michigan Crude	10 Percent Sample No. 3 90 Percent Sample No. 4 UOP Copper-sweetened
Octane No. (motor method)	82.5	81.0	82.0	65.5	67.5
Copper-dish gum, mg. per 100 ml:					
Uninhibited gasoline	10	10	17	34	36
Uninhibited gasoline plus 0.025 percent UOP No. 1 inhibitor	13	8	10	3	2
Uninhibited gasoline plus 0.005 percent UOP No. 4 inhibitor	10	5	6	3	1
Oxygen-bomb induction period, min:					
Uninhibited gasoline	35	65	55	350	370
Uninhibited gasoline plus 0.025 percent UOP No. 1 inhibitor	245	255	200	545	545
Uninhibited gasoline plus 0.005 percent UOP No. 4 inhibitor	180	360	320	580	595
Sulfur, percent	0.46	0.48	0.47	0.08	0.12
Reid vapor pressure, lb.	13.1	11.6	12.8	9.2	10.1
Gravity, deg. API at 60 °F.	69.6	68.6	69.4	61.5	62.5
ASTM Distillation in °F.:					
Initial boiling point	85	85	85	96	90
5-percent point	103	110	105	119	113
10-percent point	115	125	117	132	127
50-percent point	**207**	**209**	**207**	**236**	**229**
90-percent point	312	314	319	340	335
95-percent point	364	361	362	365	356
End point	388	389	389	378	379
Recovery, percent	97.0	97.5	97.0	98.0	98.0
Residue, percent	1.5	1.5	1.5	1.0	1.0
Loss, percent	1.5	1.0	1.5	1.0	1.0

whereas in the other two refineries the polymer product and refinery gasolines are blended and then sweetened. In general, the choice of methods used in sweetening the polymer product or a blend with refinery gasoline is influenced by the mercaptan content of the polymer gasoline. When the sulfur content of the polymer is high, in general it is well to sweeten the polymer separately. In such instances, sweetening by the use of the UOP copper-sweetening process is advantageous, because polymer gasoline of any mercaptan content responds most favorably. In general, the blending value of polymer gasolines increases with decreasing sulfur content—which lends advantage to the operation of desulfurizing the gas before charging to the poly plant.

In a polymer gasoline containing a total sulfur content of 1.99 percent, the mercaptan sulfur was 1.10 percent of the polymer. The distribution of the mercaptans follows:

	Percent
Methyl mercaptan	0.048
Ethyl mercaptan	0.321
Propyl mercaptan	0.521
Butyl mercaptan	0.178
Amyl mercaptan	0.010
C_6 and heavier mercaptan	0.020

After caustic-soda treatment the mercaptan sulfur was reduced from 1.10 to 0.082 percent, and the total sulfur from 1.99 to 1.06 percent.

A polymer gasoline of 0.46 percent sulfur and blends with other gasolines were studied from the standpoint of sweetening with the caustic soda and UOP copper salts, and their effect on the octane rating of treated products. The results are shown in Table 8.

OPERATING RESULTS

Of the five midget units discussed herein, one operates in conjuction with a reforming unit, another with a combination topping and cracking unit, and the other three with plants cracking topped crudes.

In the case of the reforming unit, the yield of 70-octane gasoline without the polymer was 76 percent. With the poly unit processing the cracked gas produced in the reforming operation, the yield of gasoline increased by 8 percent—making the total gasoline yield from reforming and polymerization 84 percent. Coincident with the increase in gasoline yield there results an increase in octane number of the blend of polymer and reformed gasoline of 1.5 to 2 octane numbers.

Table 9 gives the distillation range and octane rating of the naphtha, reformed gasoline, and 10-percent blend of the polymer and reformed gasoline.

When operating the combination topping and cracking unit, the midget poly plant increased the yield of gasoline by 1.9 percent calculated on the crude charge. Together with this increase in gasoline yield, an overall increase in octane of one number results.

In the case of one of the poly units operating on gas from topped crude-oil cracking, the stabilizer produces a debutanized gasoline—thus increasing to

TABLE 9

Reforming Data

	Reforming Stock	Reformed Gasoline	Blend of 10 Percent Polymerand 90 Percent Cracked
Gravity, deg. API	50.8	54.4	55.5
ASTM Distillation in °F.:			
Initial boiling point	180	88	88
10-percent point	256	156	156
20-percent point	272	202	203
30-percent point	286	232	234
40-percent point	297	254	252
50-percent point	309	272	268
60-percent point	324	290	291
70-percent point	343	308	309
80-percent point	366	332	334
90-percent point	395	370	373
End point	485	395	399
Octane No. (ASTM)	...	70	72
Reid vapor pressure, lb.	...	11.0	11.0

TABLE 10

Charging Stock to Selective Polymerization Unit

	Percent
Propane-propene	1.0
*Iso*butene	18.4
Normal butene	32.6
Butanes	48.0

a maximum the olefin-containing gas to the poly plant. In this type of operation, the polymer gasoline serves to increase the over-all gasoline yield from the combined operation by 3.1 percent based on the cracking stock. The octane number of the blend of the gasoline increases from 69.2 to 71.

The other two poly units are processing a lesser amount of stabilizer gas, and the increase in gasoline yield varies from 1 to 2.5 percent with an octane rise of from 0.5 to 1 number.

ROAD AND LABORATORY TESTS ON POLYMER GASOLINE

Road and laboratory tests, conducted with blends of polymer gasoline and various types of base fuels, have disclosed that such blends have a higher anti-knock value on the road than is indicated by laboratory tests. As an illustration, a typical leaded gasoline with a 69.5 octane number (motor method) was increased to 71.5 by the addition of 10 percent polymer gasoline. This blended gasoline, however, showed a road rating of 75 octane.

MIDGET POLY *ISO*OCTANE UNITS

The design of *iso*octane plants is such that it lends itself well to small units. Several midget *iso*octane units to produce 50 barrels a day of *iso*octane have been designed, and will be constructed for commercial operation. The charge to the midget polymerization unit will be a butane-butene fraction from the cracking-still stabilizer.

A typical analysis of the butane-butene fraction would show about 1 percent propane and propene, 18 percent *iso*butene, 33 percent normal butene, and 48 percent butanes. The butane-butene charge to the selective polymerization unit analyzed is as shown in Table 10.

This stock is water- and caustic-soda treated, as shown in Figure 4, before passing to heat exchangers, to remove the nitrogen and sulfur compounds present. The temperature of selective polymerization ranges from 260 to 325° F., and the pressure maintained at about 700 pounds. A flow chart of the butane-butene polymerization unit to produce *iso*octenes is shown in Figure 5. The temperature at which the selective polymerization takes place is an

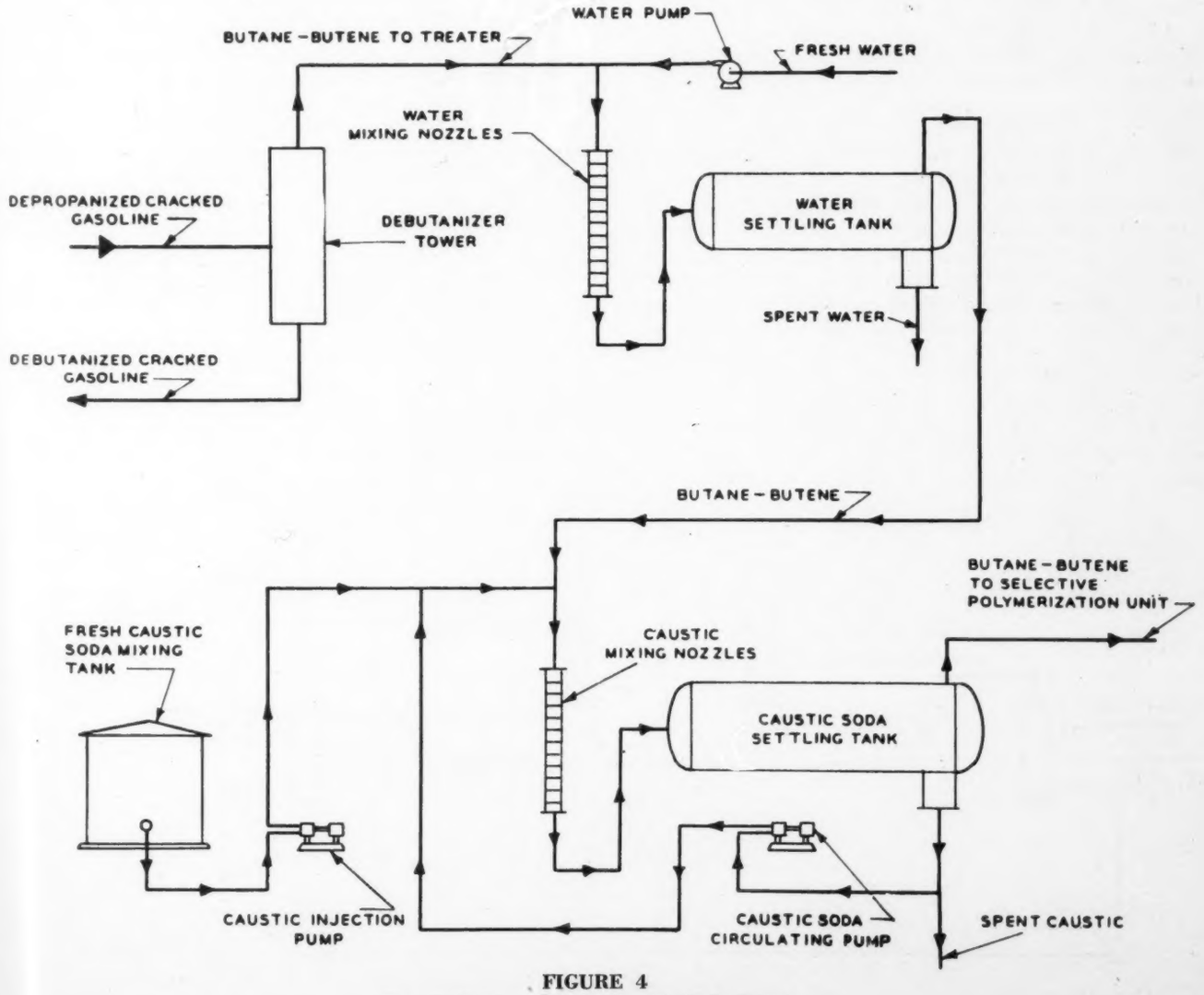


FIGURE 4

Butane-Butene—Water-Caustic-Soda Treater

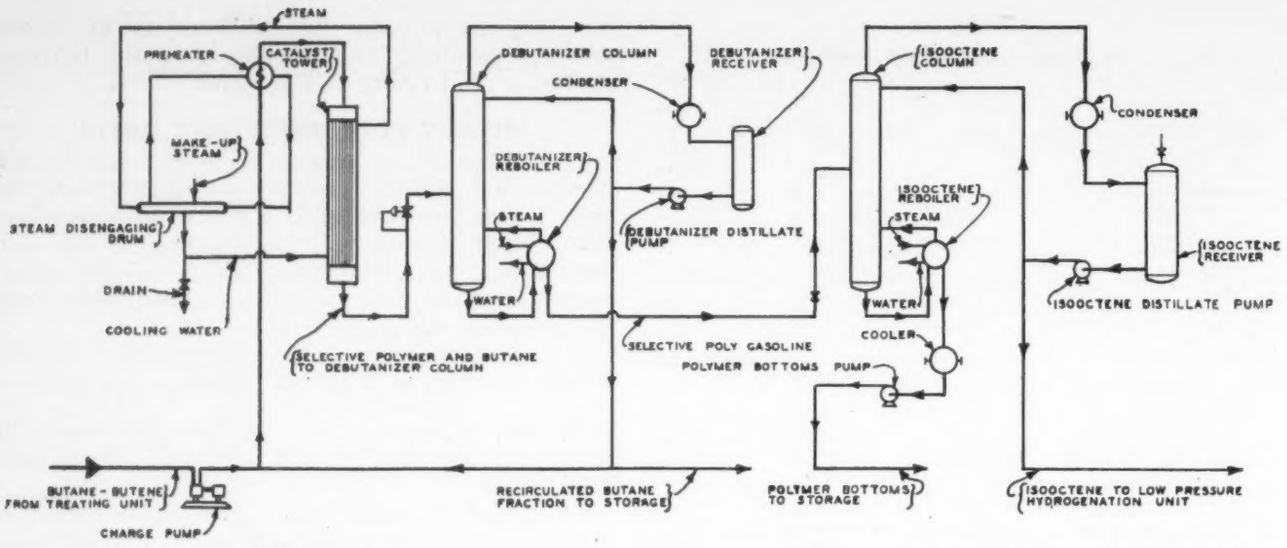


FIGURE 5
UOP Catalytic Selective Poly Unit

important factor which governs the octane rating and the yield of the hydrogenated polymer gasoline (*iso*octanes). The crude polymer gasoline made up of *iso*octenes, dodecenes, and cetenes is analyzed as shown in Table 11.

The crude polymer is distilled into an *iso*octene fraction and bottoms. The distillation and other properties of this fraction are shown in Table 14. dodecenes fraction with a small proportion of *iso*-dodecenes frection with a small proportion of *iso*-cetenes. The distillation analysis is shown in Table 15.

The conversion of *iso*butene is practically complete in the operating range of this process. The percentage conversion of normal butene increases; therefore, the polymerization ratio of normal to *iso*-butene will increase also. The yield of crude polymer increases under the same conditions while its higher-boiling components increase slightly in amount. The finished hydrogenated *iso*octanes from the *iso*octanes produced at different temperatures (260 to 350° F.) may range in octane rating from 91 to 97—dependent on temperature. The olefin conversion to polymer based on the *iso*butene present in the gas will vary from 150 to 280 percent on a mol basis. The higher the octane rating of the hydrogenated polymer (*iso*octanes), the lower the yield based upon the butane-butene charge. The percentage conversion of the olefins in the butane-butene fraction to *iso*octenes is dependent upon the octane rating desired after their hydrogenation. The yield will vary between 24 and 31 percent by volume of the charging stock given in Table 10. These polymer-gasoline yields correspond to a 30-40 percent yield by weight of the butane-butene fraction charged to the selective polymerization unit.

The *iso*octenes produced may be converted to *iso*-octanes by the UOP low-pressure hydrogenation

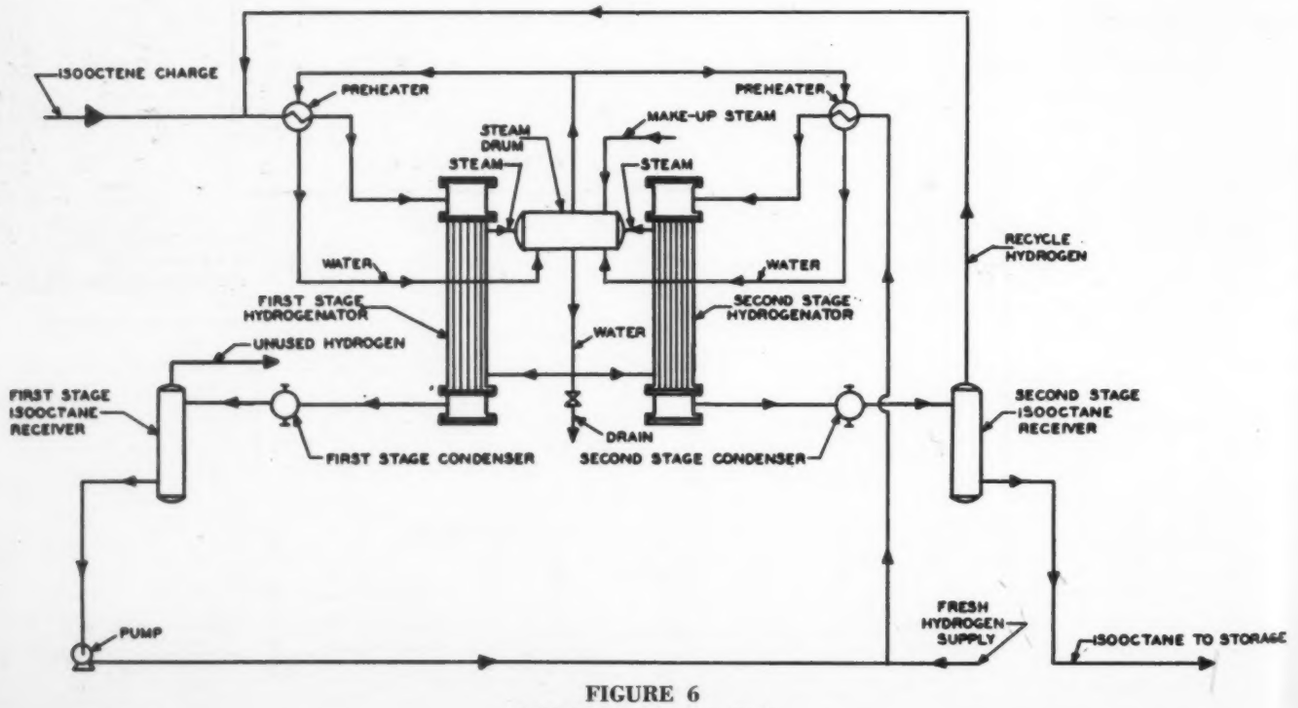


FIGURE 6
UOP Hydrogenation Unit

TABLE 11
Crude Selective Polymer

Gravity, deg. API	59.9
ASTM Distillation in °F.:	
Initial boiling point	218
10-percent point	225
20-percent point	226
30-percent point	228
40-percent point	229
50-percent point	231
60-percent point	233
70-percent point	236
80-percent point	242
90-percent point	261
End point	381
Octane No.	84.0
Reid vapor pressure, lb.	1.3
Sulfur, percent by weight (lamp)	0.005

TABLE 12
***Iso*octenes**

Gravity, deg. API	61.5
ASTM Distillation in °F.:	
Initial boiling point	200
10-percent point	221
20-percent point	223
30-percent point	225
40-percent point	226
50-percent point	227
60-percent point	227
70-percent point	228
80-percent point	230
90-percent point	233
End point	267
Octane No. (before hydrogenation)	85.5
Reid vapor pressure, lb.	2.0
Sulfur, percent by weight (lamp)	0.002
Color, Saybolt	30+

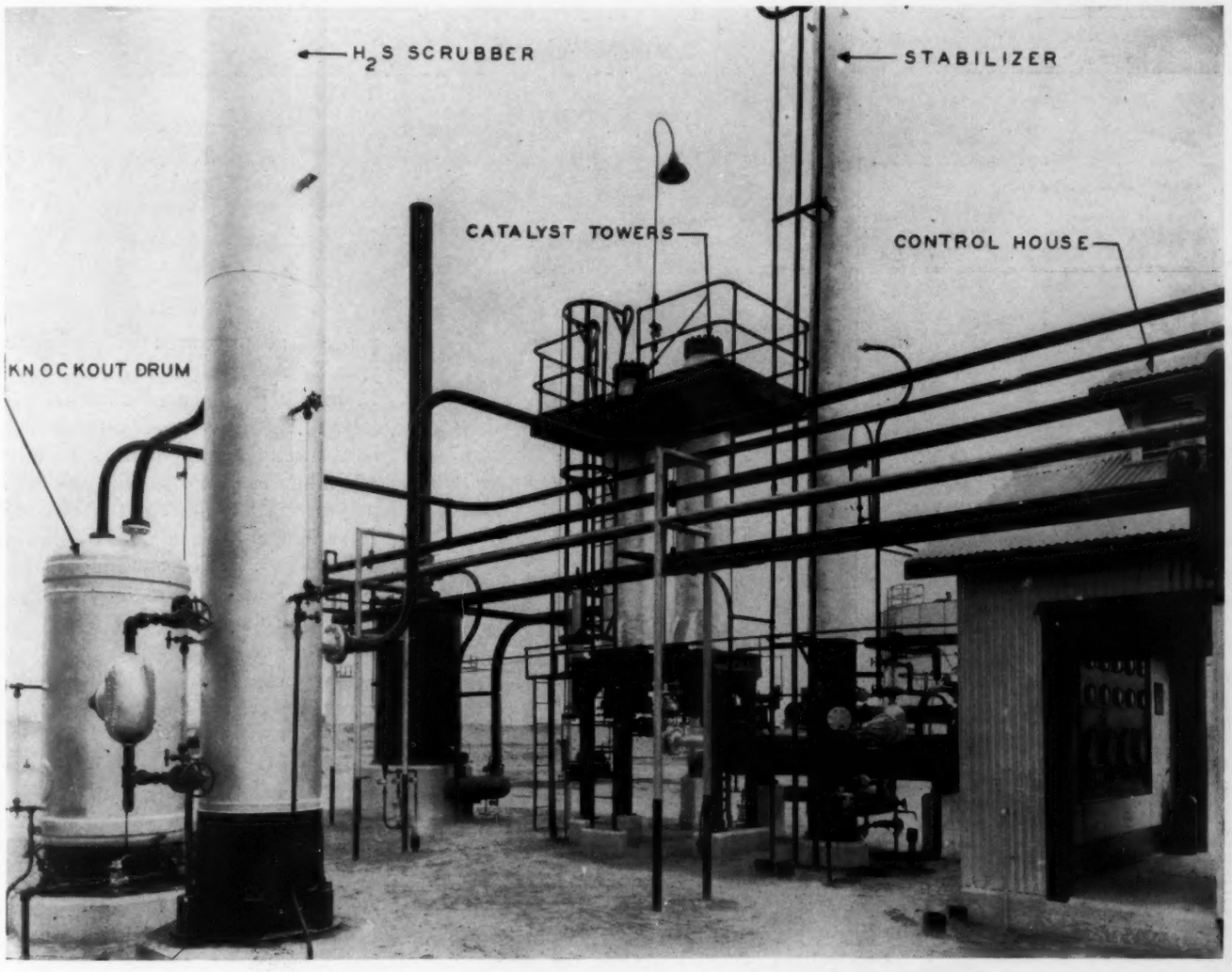


FIGURE 7
Mohawk Petroleum Corporation UOP Midget Catalytic Poly Unit

TABLE 13
Selective Polymer Bottoms

Gravity, deg. API	47.3
ASTM Distillation in °F.:	
Initial boiling point	319
10-percent point	338
20-percent point	345
30-percent point	351
40-percent point	357
50-percent point	361
60-percent point	364
70-percent point	367
80-percent point	374
90-percnet point	392
End point	467
Sulfur, percent by weight	0.02
Color	Yellow

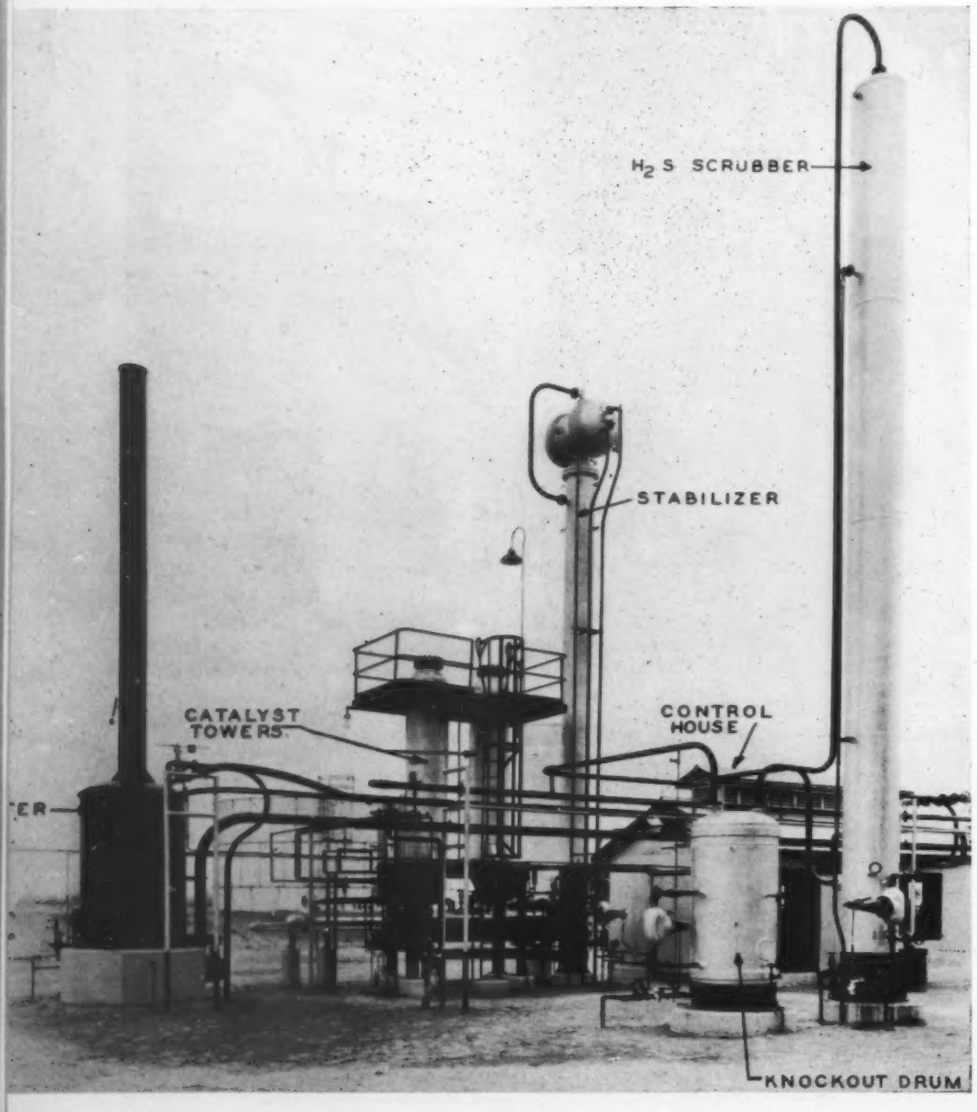
TABLE 14
***Iso*octanes**

Gravity, deg. API	66.4
ASTM Distillation in °F.:	
Initial boiling point	210
10-percent point	222
50-percent point	227
90-percent point	232
End point	254
Octane No. (ASTM motor method)	95.0
Reid vapor pressure, lb.	1.6
Sulfur, percent	0.001
Olefins, percent	0.5
Gum (copper-dish) mg. per 100 ml.	0
Color, Saybolt	30

TABLE 15

Laboratory Inspection and Anti-knock Data for High-Octane Aviation Blends

	"A" UOP *Iso*-octane	"B" Aviation Straight-run	"C" *Iso*-pentane	BLENDS			
				50 Percent "A" 50 Percent "V"	40 Percent "A" 50 Percent "B" 10 Percent "C"	40 Percent "A" 45 Percent "B" 15 Percent "C"	50 Percent "A" 40 Percent "B" 10 Percent "C"
Gravity, deg. API	66.4	66.7	95.7	66.5	68.6	69.6	68.7
ASTM Distillation in °F.:							
Initial boiling point	210	115	80	112	102	100	110
10-percent point	222	148	82	177	150	145	155
50-percent point	**227**	**185**	**88**	**212**	**204**	**203**	**211**
90-percent point	232	222	96	232	231	230	231
End point	254	250	103	253	250	249	248
Recovery, percent	99.0	98.0	99.0	98.5	98.5	98.5	98.5
Reid vapor pressure, lb.	1.6	7.2	20.1	3.8	6.2	6.8	5.7
OCTANE NUMBER							
U. S. Army Air Corps Method:							
Unleaded	95.0	74.0	90.0	84.0	84.0	84.5	85.5
With addition of 0.30 ml. tetraethyl lead per gallon	100.0	...	...	...	...	...	...
With addition of 0.60 ml. tetraethyl lead per gallon	...	...	100.0	...	...	...	...
With addition of 3.00 ml. tetraethyl lead per gallon	...	91.0	...	99.5	99.5	100.0	100+
ASTM Motor Method:							
Unleaded	95.0	73.5	90.0	84.0	84.0	84.5	85.5
With addition of 0.35 ml. tetraethyl lead per gallon	100.0	...	...	...	...	...	...
With addition of 0.65 ml. tetraethyl lead per gallon	...	...	100.0	...	...	...	...
With addition of 3.00 ml. tetraethyl lead per gallon	...	89.0	...	97.5	97.5	98.0	100.0



process at pressures of about 75 pounds and temperatures of 325° F. in the presence of a nickel catalyst. The hydrogenation reaction is also exothermic; hence, the unit is similar in design to the selective polymerization unit in the step of close temperature control by means of a water jacket around the reactor. A flow chart of the low-pressure hydrogenation unit is shown in Figure 6.

The properties of the hydrogenated *iso*octanes are shown in Table 14.

The *iso*octanes as produced are not aviation gasoline as such. They must be blended, for volatility, with aviation stock such as special straight-run or natural gasoline and *iso*pentane.

The usual procedure for preparing 100-octane aircraft fuel is to blend commercial *iso*octane with aviation straight-run gasoline and *iso*pentane, and add 3 ml. of tetraethyl lead per gallon. The *iso*pentane is used to supply the front-end volatility which *iso*octane lacks. The amount of *iso*pentane incorporated in the finished blend is usually

FIGURE 8

UOP Midget Catalytic Poly Unit

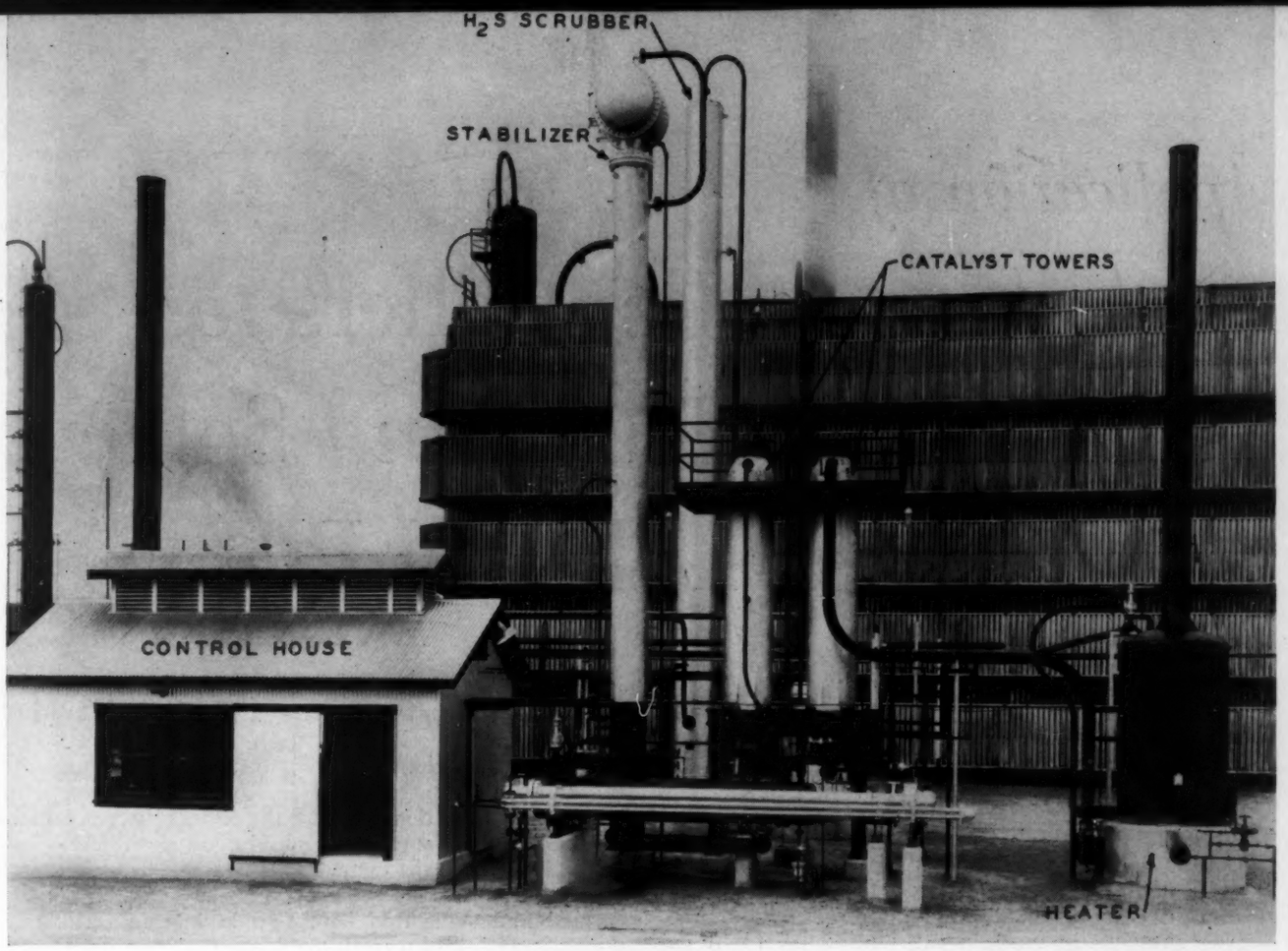


FIGURE 9
UOP Midget Catalytic Poly Unit

10 to 15 percent—depending on its availability, and also on the vapor pressures of the *iso*octane and the straight-run base fuel.

Laboratory inspection data for several such blends and for the various components used to make up those blends are shown in Table 15. Ratings by both the motor and the Army method are included. In the United States the Army method is specified, whereas the motor method is used in England, for 100-octane fuels. As indicated in the table, the Army ratings for leaded blends of this sort are usually about two octane numbers higher than corresponding motor-method values.

Based on the results given in Table 15, a commercial 100-octane (Army-method) 7-pound-vapor-pressure blend could consist of 40 percent of 95-octane *iso*octane, 45 percent of 74-octane aviation straight-run, and 15 percent of 90-octane *iso*pentane. To produce a similar blend to equal 100 octane by motor method, part of the straight-run would have to be replaced with *iso*octane; e. g., 45 percent of *iso*octane, 40 percent of straight-run, and 15 percent *iso*pentane.

Considerable discussion has centered around safety fuels for use in aircraft engines to reduce the fire hazard now present in conventional volatile-type fuels. Although the exact properties of an ideal safety fuel are not known definitely, such a fuel generally is considered to boil in the 300 to 400° F. range, and have a flash point above approximately 110° F.

The UOP selective polymerization process, used in the manufacture of *iso*octane aircraft fuels, also produces material which can be fractionated to a product boiling in the safety-fuel range. The selective poly units are operated to produce maximum yields of *iso*octenes. The yield of *iso*octenes on the polymer product can be controlled to 90-95 percent, the balance being dodecenes and hexadecenes or cetenes. However, the reaction also may produce high yields of the heavier polymers which can be hydrogenated to safety aviation fuel. The properties of this type of fuel are given in Table 16. For comparative purposes, we have included data for *iso*octane produced in the same oepration.

A safety aviation fuel can be obtained from the hydrogenated high-boiling fraction by distillation which would yield a product of 98 to 99 octane rating if desired.

Currently (May 1, 1939) midget *iso*octane units in the United States are uneconomic. These units were developed primarily for foreign countries, where special local conditions warrant their installation. Notwithstanding this situation, these midget *iso*octane units represent an appreciable potential supply of *iso*octane and safety aviation fuel in the United States.

TABLE 16

	Operation No. 1*		Operation No. 2*	
	Iso-octane Fraction	High-Boiling Material	*Iso*-octane Fraction	High-Boiling Material
Gravity, deg. API	66.4	50.5	68.5	58.1
ASTM Distillation, °F.:				
Initial boiling point	210	273	199	250
10-percent point	222	317	218	281
50-percent point	**227**	**362**	**222**	**340**
90-percent point	232	389	230	361
End point	254	450	250	390
Reid vapor pressure, lb.	1.6	<0.5	1.7	<0.5
Octane No. (motor method)	95	89	97	96

* Charging stock: Operation No. 1—one part *iso*butene to two parts *n*butene.
Operation No. 2—four parts *iso*butene to one part *n*butene

Application of Automatic Controllers *to* Solvent Refining *and* Solvent Dewaxing Lube Oil Plants

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THE comparatively recent commercial development of solvent refining and solvent dewaxing processes has been materially assisted by the use of automatic control instruments. The importance of instrumentation in modern lubrication oil plant design is indicated by the fact that instruments and control valves represented 7/10 percent of the total material cost of several recent installations. From the refiner's standpoint, the correct application of instruments in the design of solvent lube units results in accurately controlled product specifications, labor savings, low solvent losses, and smooth operation. This paper will present a general description of control applications from the standpoints of process engineering and operation, and a brief outline of selected control problems essentially unique to solvent refining and dewaxing units.

Generally speaking, several applications of instruments to each control problem are usually possible, and those presented, while not the only feasible ones, represent the control methods found most satisfactory by the authors.

SOLVENT REFINING

For purposes of definite examples of solvent refining unit control, we shall consider a furfural extraction plant, (Figure 1.). In this type unit, charge oil and furfural are continuously charged to the bottom and top respectively of a counter-current extraction tower wherein the ratio of solvent to oil and the temperatures are regulated to yield the desired products. The tower overflow or raffinate layer containing the valuable lube fractions is charged through an exchanger and heater coil to a vacuum tower for recovery of solvent. The bottom or extract layer, containing the naphthenic constituents of the charge oil, settles due to its higher specific gravity (about 1.1) and is continuously withdrawn from the extractor and charged to the recovery system. It passes through exchangers and a heater coil, thereby effecting solvent recovery by evaporation. The residue from the evaporator towers is released into a vacuum stripper, and the finished extract bottoms are pumped through a cooler to storage. The stripper condensate streams are charged to the upper section of a tower wherein separation is effected between dry furfural at the draw-off pan and a constant boiling mixture of water and furfural at the top. The overhead is condensed and the water layer, which separates out on condensing and cooling the constant boiling mixture, is charged to a small fractionator. The overhead from the fractionator is likewise condensed and separated into two layers, and the net water is withdrawn from the bottom of the fractionator to the sewer.

The automatic control problem presented by an extraction tower process is one of maintaining constant volume ratios of solvent to oil and simultaneously holding predetermined top and bottom extractor temperatures. Volume ratios are satisfactorily handled by the application of flow controllers in each stream with the orifices in the discharge lines and the controlled valves in the steam lines to direct-acting pumps or in the discharge lines of centrifugal pumps. The temperature of the top of the extractor is controlled by a vapor pressure type instrument with a bulb located in the top of the extractor and the controlled valve in the water line to a solvent charge cooler. The temperature of the bottom of the extractor is usually controlled by a vapor pressure system controller with the bulb in the bottom of the extractor and the controlled valve in the water line to a cooling bundle which extends through the extractor near the bottom. The process lag is relatively large in this case because of the large volume in the extractor with respect to the flow toward the bottom, but the application of this type of controller has been found satisfactory in practice.

The liquid in the lower portion of the extractor tower consists of a bottom layer or extract phase containing a large percentage of solvent together with the undesirable oil fractions, and an upper layer or raffinate phase consisting of the valuable lube fractions with a small percentage of furfural. The automatic draw-off of the extract layer from the tower is usually controlled by an internal type duogravity level controller. This instrument employs a weighted float which will sink in the raffinate layer, (specific gravity approximately 0.88) and float in the extract layer (specific gravity approximately 1.10). An interesting indicating instrument has been used in Europe for this service. The difference in electrical conductivity between the raffinate and extract layers

*Presented before The Refinery Division, Oil World Exposition, Houston, Texas.

is utilized to make or break circuits across electrodes placed vertically a few inches apart in the side of a vessel, thereby closing or opening circuits to a row of lights on the instrument panel.

Furfural, like most commercial solvents, boils over a very small range and at a much lower temperature than that at which most lube oils begin to vaporize and so is readily separated from the extract and raffinate phases by evaporation and stripping. The normal refinery temperature control problem is in connection with units wherein a separation of a multicomponent mixture is made by fractionation. Usually in a solvent lube unit the boiling points of the solvent and of the lube oil are so widely different that the separation approaches a single component evaporation problem. It is essential that this difference be recognized when considering the application of temperature controllers.

Reflux is generally used in solvent evaporators for desuperheating vapors only wherein reasonably close control is required for smooth operation rather than for any fractionating control. The recovery sytem top tower temperatures are maintained in the conventional manner by vapor pressure or potentiometer type instruments controlling the reflux streams.

Furnace temperature controllers are usually provided on solvent refining units to insure against overheating of the finished oil and for generally smooth operation. Furnace outlet temperature control is not required for optimum yield as in distillation and cracking units, but rather for the efficient operation of the comparatively complicated heat exchange systems found in solvent refining units. Due to the comparatively low boiling range of the usual solvents, considerable vaporization takes place before the heater outlet is reached, making it desirable to provide for two or more parallel streams, converging in a common transfer line to the flash tower. It is desirable that each of the parallel heater streams be flow controlled. Due to the high percentage of vaporization in the heater of what is essentially a single component, the temperature within the coil is not as sensitive to heat input variation as in the case of a normal pipe still. In the design of most solvent plants the direct-fired heater is used to complete the final evaporation of solvent just prior to stripping. When the evaporation is carried to this extent the outlet temperature is reasonably sensitive to heat inputs as can be seen from Figure 2. Satisfactory control can be obtained by the use of a temperature controller with the element in the transfer line and the controlled valve in the fuel sytem. In this connection it is important that the heater be properly

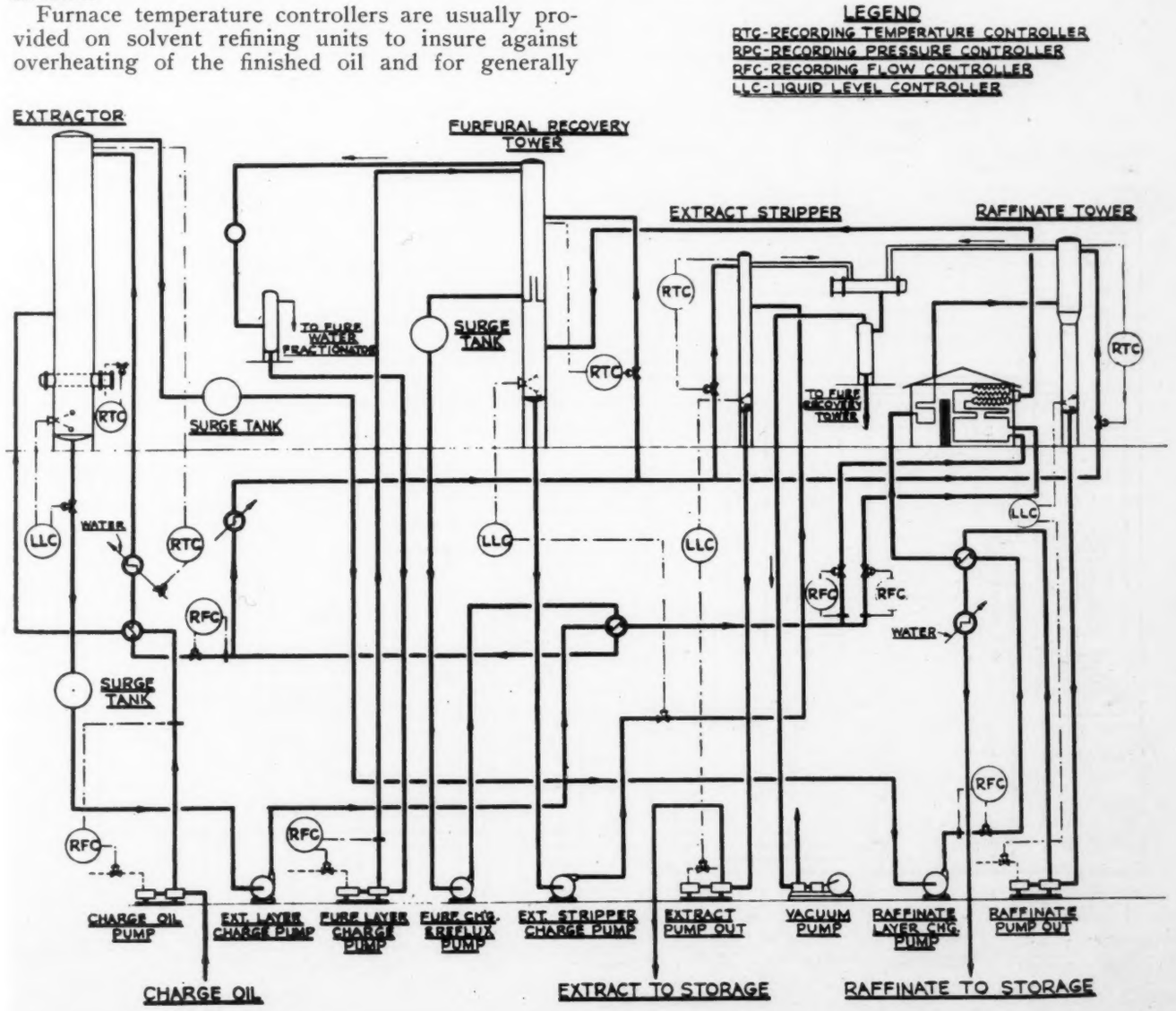


FIGURE 1

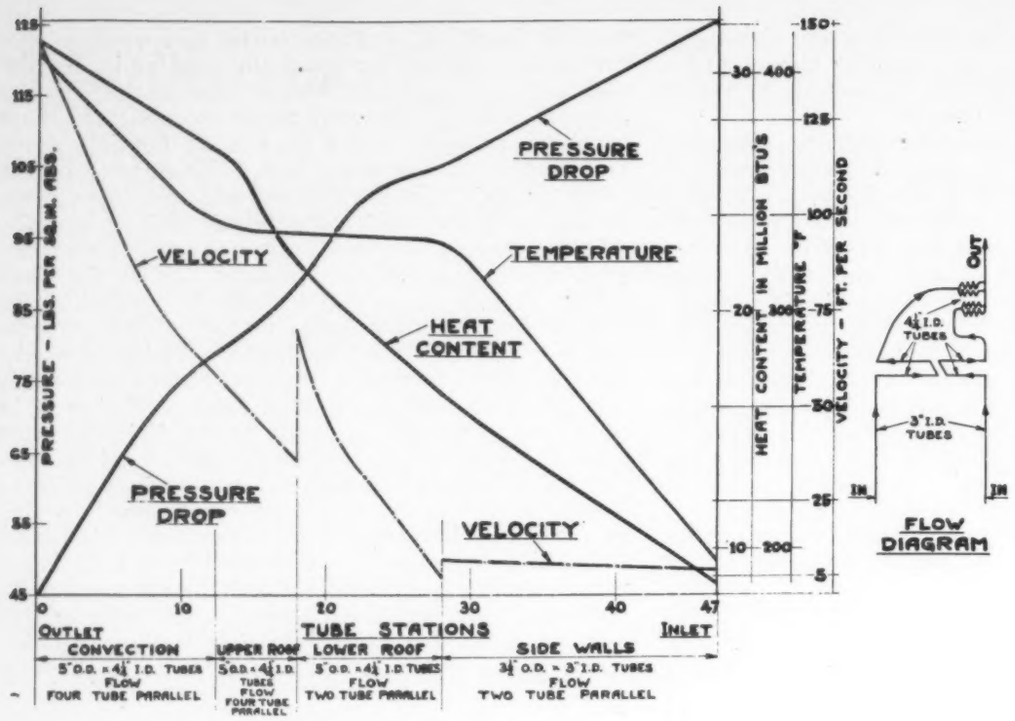


FIGURE 2

Solvent - Lube heating coil curves showing heat content, temperature and pressure relationships.

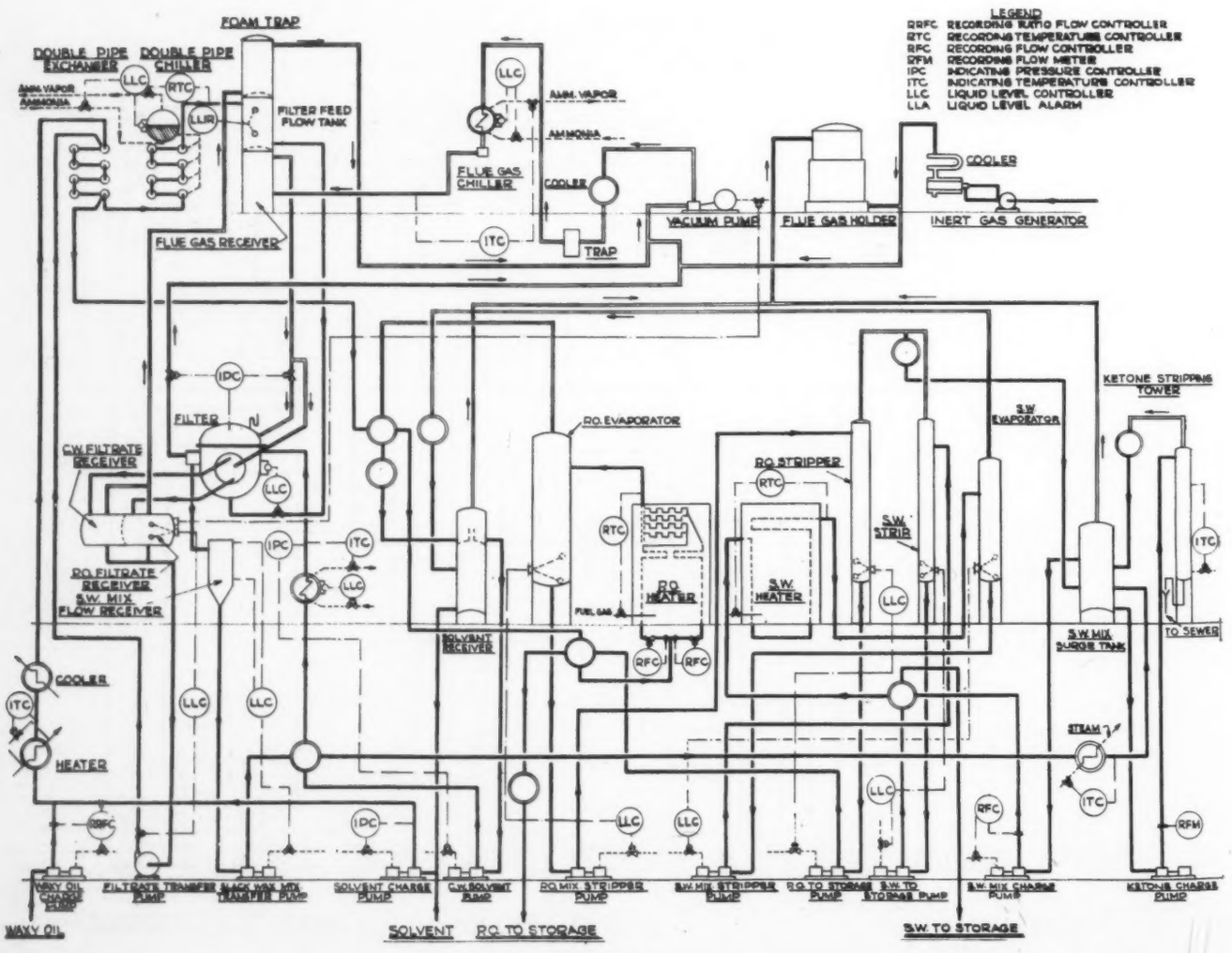


FIGURE 3

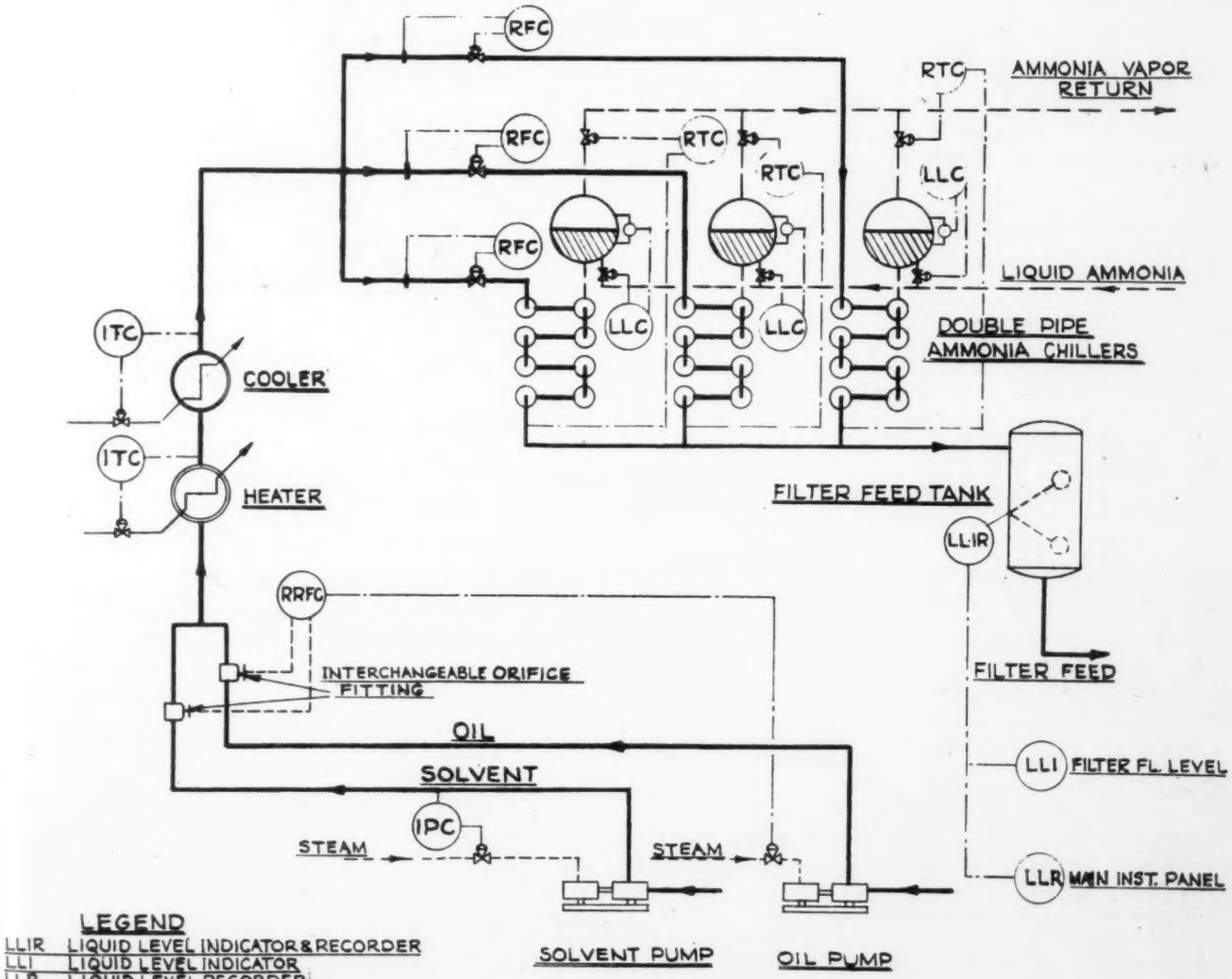
designed to accommodate the large amount of vaporization without the introduction of a temperature peak within the coil.

The use of various evaporation principles in solvent recovery systems sometimes involves the control of pressure in a system wherein the vapors are completely condensed. The pressure in such a system can be maintained by a recording pressure controller, permitting indirect control of the rate at which condensation occurs in an overhead vapor condenser. That is, the pressure controller actuates a valve in the condensate outlet raising or lowering the level of the subcooled liquid, thus blanking off a portion of the total available condensing surface, resulting in control of the tower back pressure

No special instruments are employed in furfural refining units, but several comments on general operating practice may be of interest. In units handling waxy stocks the oil flow meters are usually equipped with water seals and the leads and seal-pots are steam traced. It is desirable to provide solvent flushing lines to meters handling extract and oil-solvent mixtures, also to connect drains and vents to them in solvent drain system. In general, averaging type internal floats are used to eliminate difficulty from plugging of waxy stocks and extracts and also to insure the smoothest possible operation. This point is particularly important in this type unit because most of the flows within the unit are determined by level control from tower bottoms and accumulators. The inherent and distinguishing feature of solvent lube units that necessitates very close correlation of levels and flows is the continuous recirculation of the solvent.

SOLVENT DEWAXING

The solvent dewaxing process, which uses benzol-ketone mixtures and is the most widely adopted of the modern dewaxing methods, will be used to illustrate control problems (Figure 3). In this process oil and solvent are mixed and then chilled in double-pipe units by exchange and by direct expansion of ammonia. This chilled material is then fed to continuous rotary vacuum filters where the wax is separated, washed with solvent and discharged to a flow receiver. The filtrate is withdrawn to vacuum receivers and pumped through the double-pipe exchangers to the solvent-recovery system. There the filtrate is heated by exchange and direct-fired or steam heaters in order to evaporate most of the sol-



LEGEND

LLIR	LIQUID LEVEL INDICATOR & RECORDER
LLI	LIQUID LEVEL INDICATOR
LLR	LIQUID LEVEL RECORDER
LLC	LIQUID LEVEL CONTROLLER
RRFC	RECORDING RATIO FLOW CONTROLLER
RTC	RECORDING TEMPERATURE CONTROLLER
RFC	RECORDING FLOW CONTROLLER
ITC	INDICATING TEMPERATURE CONTROLLER
IPC	INDICATING PRESSURE CONTROLLER

FIGURE 4

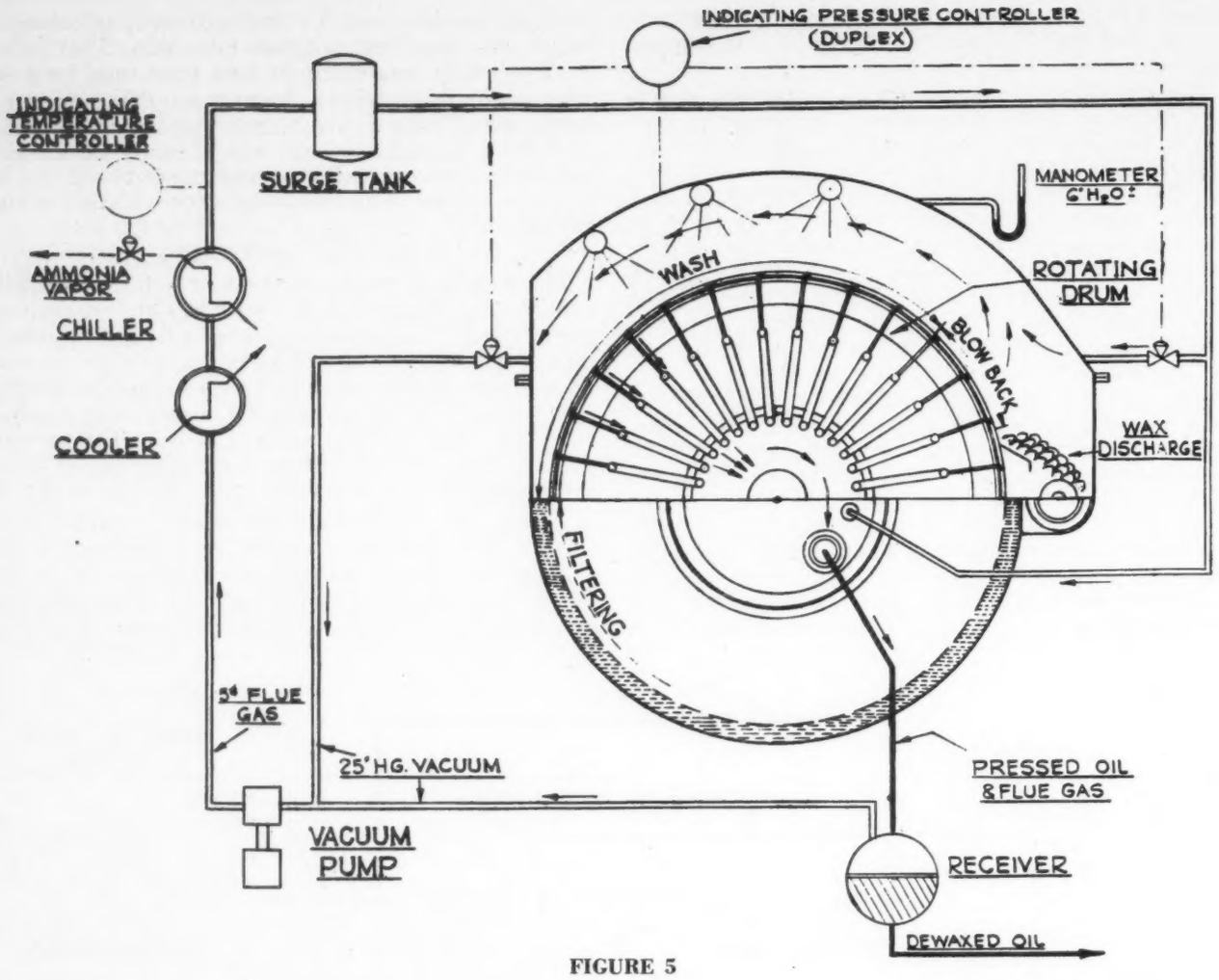


FIGURE 5

vent. The residue from the evaporators is steam stripped at atmospheric pressure.

The slack wax from the filters is heated by exchange with solvent and exhaust steam and settled to remove any water introduced with the charge oil. The solvent is then recovered from the wax in a manner similar to that outlined for recovery of solvent from the filtrate.

The overhead from the strippers is condensed and settled so that the water is withdrawn as a water-ketone layer. This water layer is charged to a small fractionator in which the net water is removed from the bottom and a constant-boiling mixture of ketone and water is taken overhead to the slack wax mix settling tank.

Accurate control of the solvent to oil ratio is essential for optimum results in solvent dewaxing. Ratio flow controllers are widely used for this service. Normally, dewaxing plants handle oils from 100 S.S.U. at 100° F. to 200 S.S.U. at 210° F., and where oils with such a wide range of viscosity are to be metered by orifice it is advisable to investigate the orifice coefficient for viscosity correction. Errors introduced by varying viscosities are reduced by maintaining line velocities at the orifice as high as possible.

If we consider the application of ratio flow controlling to dewaxing units it becomes obvious that it is desirable to have a set of orifices for each stock because the various lube stocks are processed at different charging rates and with different ratios of solvent. Further, the coefficients for each oil orifice should be corrected for the viscosity of that particular oil at its normal charging temperature. In practice this is accomplished by providing interchangeable orifice fittings for both oil and solvent charge lines and sets of orifice plates with corrected coefficients. This phase of flow-controller engineering is of recent development, and is probably of greatest value to operators of solvent-dewaxing units.

Split flow of the oil-solvent mix through the chilling units is frequently desirable from the standpoint of pressure drop and this complicates the problem of mix ratio control. Further, the rate of charge is determined in the final analysis by the rate of filtration. A satisfactory control set-up for this type system is illustrated in Figure 4. Recording flow controllers are used on the individual streams through the chilling units. The solvent charge pump is regulated by a pressure controller maintaining a constant discharge pressure and a ratio flow controller actuates a valve in the steam to the oil charge pump. The effluent from the chillers enters a surge tank from which the filters are fed by gravity. The level in this tank is recorded on the main control board as well as indicated in the filter room. The operator resets the individual stream flow controllers to maintain the desired level. Various automatic level resets have been tried in this service, but manual control has worked out more satisfactorily. The reason is that with any automatic reset device, upsets in charge rate occur as

filters are cut in or out of service. An operator, on the other hand, can let the feed tank level run low just before he stops a filter for its periodic washing and then likewise allow the feed level to run high just before starting a filter, thereby taking full advantage of all available surge capacity with minimum flow changes. Manual attention to this detail is very simple and effective in actual practice.

The problem of indicating or recording the level of the waxy mixture in the filter feed tank is met by using an internal float with air pilot. In order to be able to utilize all available surge capacity, a special type internal float mechanism is used. The ball float is attached on an arm at right angles to the pilot shaft which enters through a small tangential nozzle. Consequently, the float can travel through a 180° arc, thereby allowing a level recording range nearly equal to twice the tank diameter, if desired.

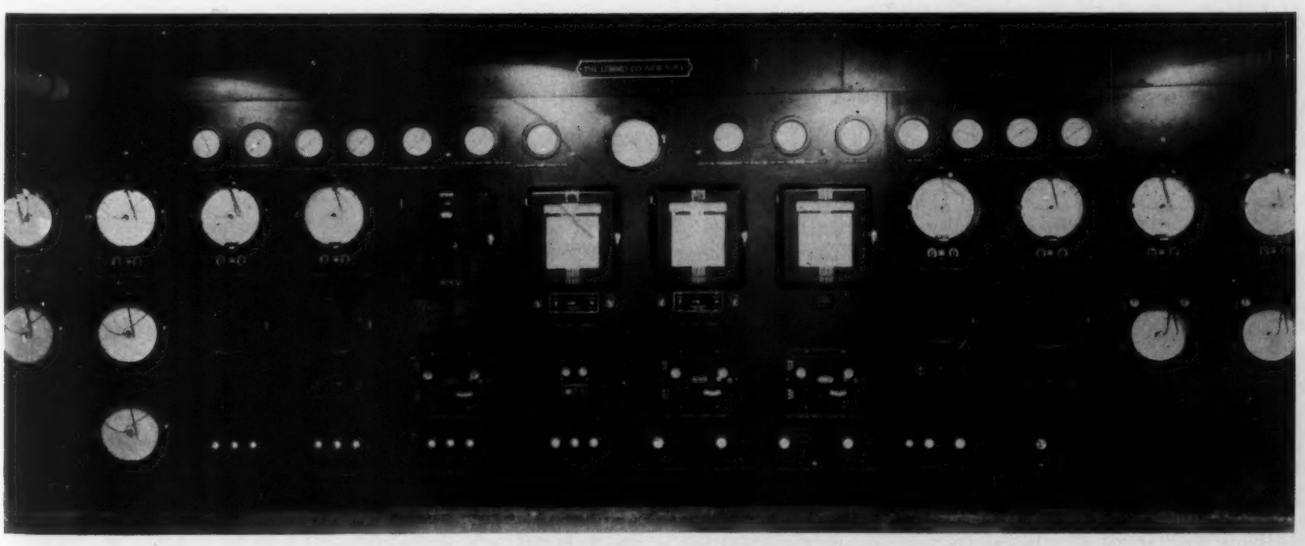
The temperature of the solvent-oil mixture leaving the double-pipe chillers is an important control point. Liquid-filled-type temperature controllers are normally used to regulate the evaporation of ammonia to obtain the desired mix temperature. This control is accomplished by installing the controlled valve in the ammonia suction line to the refrigeration plant. Thus, the final outlet temperature of the solvent oil mix is controlled by the back pressure maintained on the direct expansion double-pipe chillers. Excellent results have been obtained in approximately —25° F. service by equipping these valves with radiating finned stuffing boxes and valve positioners to eliminate valve sticking troubles caused by freezing. The important low temperatures in solvent dewaxing units are also recorded, using nickel resistance type elements. Particular attention must be given to the location of thermowells in systems containing precipitated wax so as to avoid accumulation of wax on the thermowell. Experience indicates that the most satisfactory location is perpendicular to the flow in a straight pipe run rather than in elbows or corner fittings.

For years, it has been standard refrigeration practice to use a very simple direct-acting float-chamber type level controller for regulating the level in ammonia accumulators on chilling equipment. These have been abandoned in the more recent design of solvent dewaxing units because of their tendency to stick and their lack of control characteristics. In their place, standard kidney type floats and air-actuated diaphragm valves are used for holding ammonia accumulator levels. This equipment gives excellent results in this service. The level controls in this case simply maintain the liquid ammonia level in the chillers while the ammonia vapor back-pressure valve actually controls the outlet temperature.

The solid slack wax cake from the filters is discharged by a screw conveyor into a flow tank from which it is pumped to the solvent recovery system. The problem of controlling a level in these tanks is very difficult because of the semi-solid nature of the material. Satisfactory results can be obtained by using a manometer-type level controller with a carefully regulated continuous "bleed-back" of solvent to keep the lead lines free from wax. The conveyors dump the wax cake into the top of the receivers, and consequently internal float controls are difficult to employ.

The feed to the rotary vacuum filters is by gravity from an overhead tank and is regulated by level controllers on the individual filters. Kidney float-type levels with air pilots are used, and a continuous cold solvent wash is employed to keep the kidney free of wax. The floats are especially designed so that solvent can be introduced around the float shaft to keep it free from wax and also to minimize level disturbances. Radiating fins are provided to prevent the shaft from freezing. The valves used to control the feed of the cold waxy mixtures are of the butterfly type, equipped with radiating fins and connections for periodic flushing with warm solvent. This system of filter level control operates in completely satisfactory manner. The butterfly valve is especially adapted to this service where a low pressure drop is allowed, due to the low head gravity feed and because the flowing mixture has a tendency to plug at any point where there is an appreciable resistance to flow. In this particular application tight-shut-off is not necessary.

The rotary vacuum filter used in this process for the separation of precipitated wax consists of a sectionally divided cylindrical drum which can be revolved inside a closed vat. The chilled oil-solvent



Main instrument panel in a 4400-barrel-a-day benzol-ketone dewaxing unit

Elevation at Rotary Filters in a 4400-barrel-a-day benzol-ketone dewaxing plant.

mixture in the vat is maintained at a level corresponding to about 60 percent drum submergence and the filtrate is withdrawn through a rotary valve. This rotary valve consists of a multi-port plate which rotates with the filter drum, sealed against a machined fixed head through which the dewaxed filtrate, the wash solvent and a portion of the inert gas are withdrawn intermittently as the drum rotates. The wax cake formed on the drum periphery is discharged by blowing the inert gas through the rotary valve. The instrumentation of this portion of the plant must be applied to operate in a supplementary manner with respect to the rotary valve.

It is desirable to maintain a constant low-pressure blanket of chilled flue-gas over the filter drum and this presents the most interesting related control problem because of the variable and intermittent factors involved. The primary circulating flue gas circuit as shown in Figure 5 consists of a vacuum pump taking suction from the filter hood through the filtrate tanks and discharging through coolers, chillers and surge tank at approximately 5 pounds gauge pressure back to the filter hood through the rotary valve. The variables affecting the circuit are first the quantity of gas withdrawn through the cake into the filtrate system and second, the amount of gas blown into the hood through the blow-back system used to discharge the wax cake. Both of these items are beyond the usual control of the operator, since they are primarily a function of the wax cake characteristics.

Until recently the method of maintaining a constant pressure of chilled flue gas in the filter hood was to use a circulating blower and a multiple diaphragm low-pressure-gas regulator. A simpler, more positive and sensitive method of control has been developed recently. The application consists of a duplex indicating controller with the hood pressure acting as the control point and two diaphragm valves actuated from this single instrument. One diaphragm valve is located in the pressure gas line to the hood, and opens to admit gas when the hood pressure falls below a control setting. The other controlled valve is in a line from the hood to the vacuum pump suction and opens to release gas when the hood pressure exceeds a control setting. The control settings which operate the two valves can be changed in the usual manner but they always differ by 1-inch water pressure so that when the indicating pointer is between the two control settings, both valves are closed. The control element used is a spring-loaded inverted bell manometer. It has been found that this type of control contains sufficient dampening effect to prevent noticeable hunting, and on the other hand is sensitive within about 1½-inch water pressure.

The application of control instruments to dewaxing unit solvent recovery systems is similar to that outlined above for solvent refining units. The ketone fractionator charge is flow controlled, and the stripping steam to this tower is automatically regulated by a vapor pressure type controller to maintain a fixed temperature in the stripper about one third down from the top. The feed enters on the top deck and by varying the temperature control setting, it is possible to vary the stripping action as desired.

In general, internal type floats are used in all wax vessels. Wax flow meters are provided with solvent seals, and the lower portions of the seal pots containing the wax are steam traced. An interesting type of Venturi meter has been reported for handling slack wax with various percentages of solvent. The entire inside is chromium plates and provided with solvent wash connections both upstream and downstream of the throat.

Snap action shut-offs are sometimes used to cut off the fuel to direct-fired heaters in event of excessive tower pressures as a safety measure and to avoid the loss of solvent through relief valves. This type of instrument must be manually reset after the upset condition has been corrected. Emergency high level float shutoffs are occasionally installed on filtrate receivers to shut down the vacuum pump in order to prevent damage by liquid entering the cylinder. In general the application of instruments as safety devices is an important feature of modern instrumentation.

It is desirable to distribute the control instruments in a solvent dewaxing unit into three separate groups. In addition to the customary use of a main pump-room control panel certain instruments should be grouped in the filter room, and all chilling controllers should be separately grouped convenient to the refrigeration operator.

From a consideration of the few control problems described above, it is obvious that the successful commercial development of solvent refining and dewaxing processes depends to a large extent on the proper application of automatic control. The instrument manufacturers deserve a great deal of credit for their assistance in the application of controls and for their development of new instruments to meet problems that have arisen.

PRACTICAL USE *of* Engineering Factors *Governing Selection of* AUTOMATIC Control Equipment

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THE underlying principles governing automatic control of continuous processing have been developed mainly in the last 10 years. Along with the evolution of these principles there has been a parallel development of control equipment, the operation and characteristics of which have been based on the successively established principles.

At the present time no major development of underlying principles is in sight and, as a consequence, no forward development of control-equipment characteristics can be expected at this time.

Basing the statement on this premise, it is probable that the next major step in control technology is in the field of proper application. Sound development along this line must depend on a departure from present crude methods of analyzing control problems, which are largely empirical. Possible starting points of quantitative methods for applying automatic controls to refinery equipment are given.

The purpose of this paper is to bring to the attention of the refining industry the need for a sounder method of applying, selecting, and installing automatic control equipment for refining processes. It is desired to promote discussion and thought along these lines.

This paper was presented at Ninth Mid-Year Meeting, American Petroleum Institute, at the Roosevelt Hotel, New Orleans, May 17, 1939.

AUTOMATIC control in the oil-refining industry has developed from comparative insignificance to its present important position in approximately 10 years. This growth has been characterized by an intermittent development in the underlying engineering fundamentals involved in the design and application of automatic control equipment. Ten years ago few engineers knew of what is now generally termed "throttling range" (which means that portion of the travel of the actuating unit that is used to move the control valve), and still fewer had any conception of its significance. The control equipment which was then available reflected this condition; and in addition its mechanism, judged by present-day standards, was designed and executed crudely.

With the development and understanding of basic engineering principles of automatic control, a parallel change in design and manufacture of equipment took place. Equipment became available which incorporated previously-unheard of amounts of throttling range—also the attendant mystery of the day, known as "reset," by means of which the instrument is returned slowly to the point at which it is set to control, regardless of the position of the control valve. Various modifications and amplifications of the different phases of the apparatus made their appearance as the full nature of the underlying principles became understood better. These included so-called "averaging" level controls, which use well over 100 percent throttling range; combination installations, in which one controller accomplished its purpose by working through a second controller; etc. In all of this activity the one underlying factor which appears most obvious from today's backward glance into the past is that the equipment apparently followed the development of the fundamentals. Theory led; equipment and application followed; and another forward step was accomplished.

SITUATION NOW EXISTING

This formula for the advancement of the art of automatic control, which has worked so well in the past, apparently is not at work today. The last few years have seen a consolidation of previous advances in basic principle, as exemplified by the characteristics of the equipment now offered by the control industry, and it is fairly evident that the industry is not in a position to offer users any basic advance in underlying principle at this time. Discussions with a representative group of engineers of both suppliers and users have brought to light no hint of any expected forward step in underlying principles that might affect basic design or application.

It is probably correct to say that, with minor exceptions, the past advances in the art of automatic control have been due mainly to the activities of suppliers. It may be true that users—and particularly the oil industry—pointed out the difficulties to be overcome, but the actual working out of the equipment to meet the problem generally was left to the manufacturer of control equipment.

If the supplier now has reached the limit of his ability to create advances in the art of automatic control, it will be necessary for the users—the oil refiners among them—to take a few steps to continue progress beyond the point now reached. Such steps are easily within the power of the oil industry.

It is difficult, if not impossible, to maintain that refiners have made or now make the best possible use

of the automatic control equipment that has been, and is, available. Everyone concerned with control equipment probably could point out instances in which such equipment is installed unintelligently; or if it has been installed correctly, it is mistreated, given little or no intelligent service; is ill-adjusted, if adjusted at all; and, as a consequence, one or more such conditions are condemned to the junk heap without a decent trial.

However, it is not the purpose of this paper to deal with these well-known problems of installation and maintenance. The point to be dealt with has to do with the application of the control equipment to the process unit, with the methods for specifying, selecting, and purchasing automatic control equipment.

Methods found in use for specifying and purchasing control equipment are sometimes little better than the methods outlined above for maintaining this equipment. When this occurs, the thing that causes the greatest wonder is that any control equipment other than a pressure gage and thermometer find eventual continued use. That some controls are continued in use, despite poor application and maintenance, bespeaks the practical necessity for controls in the present-day refining of oil.

It is almost universal practice in applying automatic controls—or in "instrumenting a job," as it is frequently termed—to use methods which are wholly empirical and qualitative at best. Decisions on what to control and how to control it are based on past experience on similar jobs, together with a fairly clear knowledge of what major conditions on the unit should be maintained constant. Sometimes a comparison is made of those factors that influence controllability. More frequently, no such comparison is made; and in many cases it is undoubtedly true—judging from results—that the factors that influence controllability are not known by those responsible for applying and selecting the equipment.

IMPROVED METHODS REQUIRED

It would advance the proper use of automatic controls immeasurably—with resulting concrete advantages to oil refiners—if simple rules based on quantities, rates, sizes, capacities, etc., would be formulated that could be used as a sound basis for applying automatic controls. No one can use such a quantitative method today, because the rules have not yet been worked out. Certain individuals may know within limits what factors are important; but if anyone has a sure, sound quantitative method—or methods—for knowing "what" to use "where," he apparently has kept it well hidden.

Several papers have been published recently which indicate that considerable thought is being given the problem.[1, 2] A general comment that seems to apply to these papers is that, in approaching the problem, a large amount of fairly complex mathematics became necessary. It is quite possible that the mathematics can never be eliminated from the analysis of a general problem. However, it cannot be disputed that such mathematics are beyond the use of the average person involved in the application of controls. In this case, what will be needed will bear the same relation to such complex mathematics as a set of navigation tables bears to the astronomical calculations which are their basis.

Much more rapid strides along the path to a work-a-day quantitative basis of analyzing control problems are needed. Unless this goal is reached, automatic control is not going to "grow up" and achieve its full stature, but will continue to tag along as it is doing now.

SUGGESTED STEPS

It may not be possible to develop quickly the underlying principles involved in a quantitative solution to a general problem. However, it should be possible to arrive at the limits to which various factors in an oil-refining problem must be held in order to get results. Thus, it is submitted for consideration that the following steps can, and should, be taken by refiners as the opportunities are presented; and it is certain that opportunities to accomplish these steps are presented periodically.

1. Engineers concerned with application, installation, and maintenance of control equipment at the very least must realize what factors are involved in the various control problems, and make use of this information. Management can, and should, be critical of the reasons given for choice and location of equipment.
2. Reasoning used on a job—such as reasons for choice of level control when resetting a flow controller—should be checked in the field when the process unit goes on stream. This will pay dividends in preventing repeated mistakes.
3. More important than either point (1) or (2) and a definitely quantitative step would be: Testing of process units to determine extent of factors that influence controllability, reasons for failure of control equipment, reasons why other controls give no trouble.

   This is inherently the refiners' job, and would be beneficial mainly to refiners. Much wordy discussion by control engineers could be eliminated with a few hard-and-fast facts determined by field tests.
4. In addition to the first three points, it should be mentioned that some control-equipment manufacturers have taken the trouble to build and set up dummy apparatus to try to duplicate the control characteristics of large process units. It would involve no great expenditure of time, effort, nor money to compare actual units or portions of units to this dummy equipment, and possibly prove the dummy suitable for working out control problems—much as a pilot plant works out process problems prior to the building of a commercial unit.

Two concrete examples, showing the type of problem that would be benefited, are given—with the thought that they might crystallize comment. These examples are merely possible starting points in applying quantitative reasoning to refining problems. It is not claimed that the solutions are precise—they are not. They are put down for what they are worth—to show how some of the control problems may be approached on a fundamental basis that can be repeated and checked from job to job in an effort to find the limiting points of the various factors involved.

EXAMPLE No. 1

Problem

In Figure 1, indicating a common steam-heated kettle-type reboiler on a stabilizing column, what type of level control equipment should be obtained?

To take a specific and concrete job, the following data is assumed:

Rate of flow from reboiler to storage, 6000 gallons per hour.
Diameter of reboiler shell, 36 inches.
Normal level-center line of float cage, center of shell.
Length of shell beyond weir, 24 inches, expected.

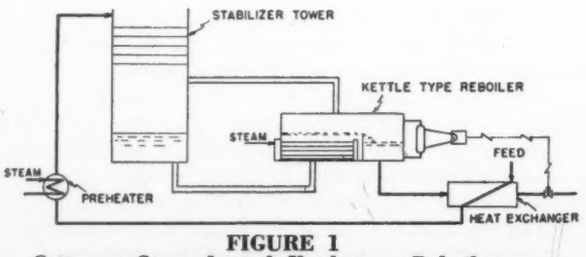

FIGURE 1
Common Steam-heated Kettle-type Reboiler on Stabilizing Column.

Solution

The outflow from the reboiler should be "stable," in order to prevent sudden fluctuations in heat transferred in the exchanger, which would in turn cause "upsets" in the tower conditions.

Statements similar to the following frequently are encountered in control papers and in discussions: "Averaging or wide-throttling level control, which is designed to produce this stability, cannot be of value unless there is sufficient capacity between the level limits on which the controller can work." No information has ever been uncovered in the literature or discussions to indicate how much capacity is "sufficient" to obtain "stable" flow.

A little thought on this subject brings out the point that the problem should be reversed, somewhat as follows: What length of reboiler beyond the weir is necessary to produce stable flow? This problem has been solved fairly successfully as follows:

With a 100 percent throttling-range setting on a level controller having a 10-inch float travel, experience indicates that a rate of change of level of 1 inch per minute with the valve operating well off its seat (50 to 75 percent of lift) gives a very satisfactory flow line. A rate of 2 inches per minute is set up more or less arbitrarily as a limit.

It is necessary to assume a figure on percentage change of inflow to the reboiler that will be considered as the maximum upset the automatic control is to compensate for. Taking this figure at 10 percent of design flow, the solution is:

Take 34 inches as the average width of shell between level limits:

$$\frac{34 \text{ times length (in inches) times } 2}{231} = \frac{6000}{60} \text{ times } 0.1$$

$$\text{Length} = \frac{(6000)\ (0.1)\ (231)}{(60)\ (34)\ (2)} = 34 \text{ in.}$$

Thus, the length of the reboiler shell beyond the weir should be about 3 feet to insure enough capacity to obtain stable outflow with a 100 percent throttling-range level instrument having a 10-inch float travel.

EXAMPLE No. 2

Problem

In Figure 2 a fractionating column is shown—together with the overhead circuit consisting of a condenser, reflux drum and pump, pressure controller, and

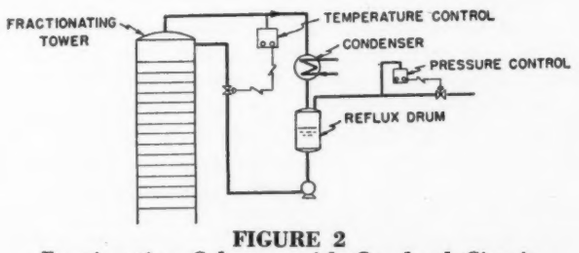

FIGURE 2
Fractionating Column—with Overhead Circuit.

a tower top-temperature controller. It is known that most towers in a refinery are controllable with a setup of equipment as shown. Other towers cannot be so controlled, and the temperature controller in the vapor line often must be dispensed with. What is the basic reason behind this frequent failure of the process to respond to automatic temperature control? At what point should automatic temperature control be dispensed with?

Solution

A consideration of the locations where temperature control has been tried indicates that: 1, towers having very complex overhead products, such as cracked distillate, finished gasoline, etc., are generally among the controllable examples. Conversely, towers having few components in the overhead vapors, such as stabilizing columns, butane towers, etc., are generally among the examples in which vapor-line temperature does not respond to automatic control.

If the case of the tower with few components is carried to the extreme—in which case the overhead vapors would have a single constituent—it is known that automatic control of top-tower temperature is impossible. For such a situation the tower pressure dictates the temperature of the overhead vapors, the same as the pressure of a steam boiler (without superheater) determines the steam temperature. It is seen readily that automatic temperature control in such a situation is quite hopeless and, of course, should not be attempted.

An example which is apparently near the border line of such control is that of the usual cracked-distillate stabilizer. Automatic temperature control of the vapor line of such a stabilizer generally is avoided today, as the result of experience.

The overhead vapors of such a stabilizer usually are composed of but four to six major constituents. Certain experiences in the field indicate that major differences in design, capacity, percent load, etc., have little effect on the controllability; but that location of the sensitive element (changing from top section to several trays below top section) has profound effect. It is thought that the common denominator of this problem that concerns controllability is temperature gradient along the tower at the point of control.

Although more information will be required to work out a true quantitative solution to this problem, it is of considerable benefit to have an example such as a stabilizer to use as a border-line case. It is at least a known point which can be used as a basis for further study.

In arriving at a conclusion for a specific job, it has been found quite satisfactory to compare the temperature gradient along the upper section of the tower in questions with the gradient along the upper section of a stabilizer. Analysis of several examples in which temperature control failed shows the gradient to be less than in a stabilizer.

CONCLUSION

A major need exists today for sound, practical rules to guide application of automatic controls to refining equipment. These rules must be simple, and must be based on rates, sizes, capacities, and other quantitative information.

Such rules are notable today mainly for their nonexistence, despite a fairly wide appreciation of their need in modern refining.

It is hardly possible for one individual or one organization to work out more than a few of the basic rules. Tests of equipment and a correlation of field information are required. It is hoped this paper will stimulate comment and discussion along this line, and thereby lead to the performance of the test work and the correlation of the field information.

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Control Practices *in* Processing Natural Gas

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THE purpose of this paper is to give a short review of current practice in the application of automatic controls to various natural gas processing operations; primarily within the range of our experience. It should be emphasized from the beginning that control schemes are susceptible to perhaps more variation than any other element of a processing plant, and that there may be several equally good solutions for any particular control problem. It is, therefore, understood that when we get down to discussing particular operations, the schemes presented are merely ones which we happen to have used satisfactorily; they are not the only feasible schemes, nor are they necessarily the best possible ones.

GENERAL PRINCIPLES OF CONTROL

The following simple principles seem so self-evident that there should be no need for repeating them here; yet we have all seen control applications where the neglect of one or more of them has led to faulty performance, or waste of money, or both. These principles are:

1. Control only essential functions.
2. Use the fewest devices which will secure the desired control.
3. Match the refinement of the control to the accuracy required by the process.

As an example of violation of principle 1, we can all remember that in the early days of high-pressure natural gasoline stabilizers, for making a constant vapor-pressure product, attempts were made to control the column top temperature as well as the base temperature. In time it became clear that the top control was theoretically as well as practically wrong. It is now usual to employ a top-temperature indicator or recorder for operating information only. Another example in connection with stabilizers is the fairly common present use of an automatically controlled heater on the feed. Theoretically, the accuracy of fractionation is improved by feeding at a constant temperature; but in practical operation we have never been able to justify this control—at least with normal natural gasoline plant stabilizers. Probably this is accounted for by the fact that the variations in heat input through the feed when this control is omitted are insignificant as compared with the heat load represented by the reflux and stripping vapor within the column.

Still another example of unessential, and perhaps uneconomic, application of control is the recently proposed use of a speed governor, responsive to suction pressure, on a vapor-recovery system compressor. The gas-engine-driven compressor unit which is to be used carries a constant-speed governor as standard equipment; further, the pressures in the atmospheric storage tanks from which the vapor is to be taken must be individually controlled by sensitive regulators anyway; hence the control system is complete without the variable-speed governor. The only advantage of the special governor is that it would allow the compressor to operate at somewhat reduced speed most of the time, and would thus probably save some wear in the unit. Whether this saving would prove enough to offset the high first cost of, and possible operating trouble with, the special governor is doubtful.

Violations of principle No. 2 are comparatively common, because most of us see the complicated way of handling a control problem first, but must spend time and thought to eliminate the unnecessary elements and work the system down to its essentials.

An old example of violation of the principle of simplicity, of which I have seen several instances, was the use of individual steam-pressure-responsive fuel governors on boilers operating in parallel. Obviously, it was impossible to get any two governors to respond exactly together; hence one or two boilers with the most sensitive regulators would try to carry all the load while others idled. The obvious correction was to use a single regulator to control the fuel to all the boilers, so that the firing in all changed together.

Another detail frequently seen in absorption plants illustrates this principle particularly well. It is often required to weather dissolved gases from a stream of absorption oil, under a pressure just high enough to permit delivering the oil to some piece of apparatus further on in the cycle. One way to do this is to determine what maximum pressure will be required for delivery of the oil under the worst conditions, and to put a back-pressure regulator set for this pressure on the vapor leaving the tank, thus fixing the weathering pressure; and to provide a level controller for handling the outflow of oil. The simplified scheme requires only a level controller, with its valve on the vapor leaving the tank. This scheme not only eliminates the superfluous back-pressure regulator, but also permits using the lowest possible weathering pressure at all times.

* Presented before Refinery Division, Oil-World Exposition, Houston, Texas.

Principle No. 3 simply says, "Don't use a highly-refined instrument or control in preference to a simple one, unless the extra first cost and maintenance of the refined instrument will pay out."

For example, we have all seen fully compensated flow recorder-controllers, suitable for extremely delicate and accurate control under steady flow conditions, used to control the speed of a simplex direct-acting steam pump. On account of the pulsating, intermittent flow the recorder will give only a rough relative idea of the actual flow rate, and the process lag compensating feature of the instrument never has a chance to operate. Obviously, the simplest type instrument would serve equally well, and would show a material saving in first cost and maintenance.

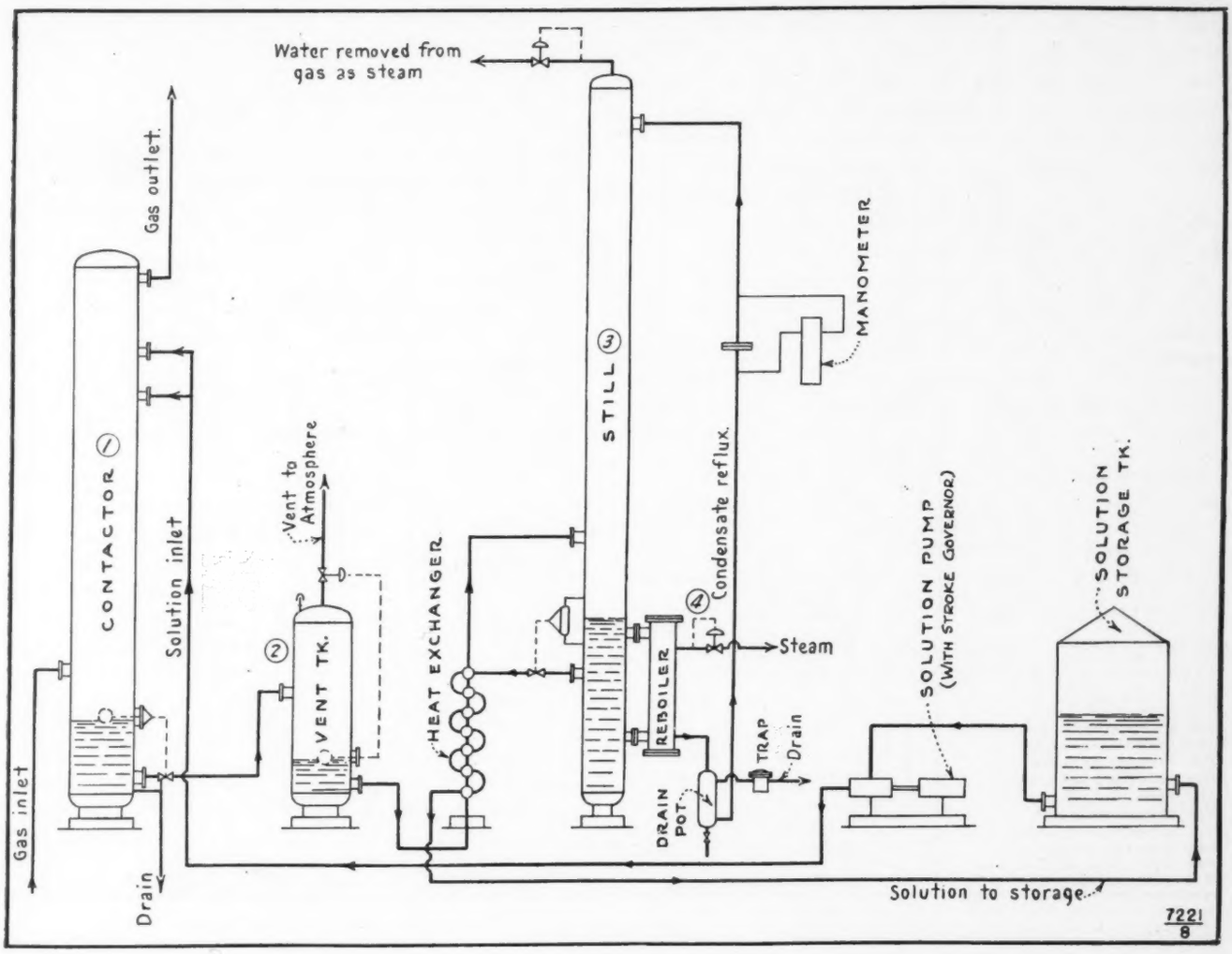


FIGURE 1

FLOW SHEETS

The first step in designing a control system for a process is the laying out of the apparatus and connecting piping in the form of a diagram or "flow-sheet," so as to indicate the functions of each element, and its functional relation to the others. All process controls are indicated on this diagram; study of the control system in this form will almost always develop ways of simplifying the system as originally conceived. Our own experience has shown that just as much time as possible should be spent in studying the control system at this stage, since such study almost always pays big dividends in producing a simpler and more satisfactory plant, as well as in reducing costs. I recall one recent case involving the design of an automatic butane-air mixture gas standby plant for a large city gas system in which we had seven essential controls in the first flow-sheet; in the final design this was reduced to three, without sacrificing any operating advantages.

In laying out a flow-sheet for preliminary study, the "straight line" arrangement, illustrated by Figure 1, is easiest to follow. The essential pieces of apparatus are drawn in a row, arranged in sequence according to the main flow; in this case, the flow of the processing solution. Interconnecting piping naturally arranges itself simply; the longer runs between pieces of apparatus which are not adjacent are dropped down below the line of apparatus. For complicated processes this arrangement may become awkwardly long; two or more "straight lines" can then be placed one above another, as illustrated by Figure 2.

It is understood, of course, that the flow-sheet arrangement will bear little or no relation to the actual plant layout arrangement. The whole purpose of the flow-sheet arrangement should be to make the diagram as simple to read as possible, to facilitate study looking toward simplification.

ACTUAL CONTROL APPLICATIONS

In order to bring out some of the points regarding selection of controls which we have learned from our own experience, I will follow through two actual plant flow-sheets, covering somewhat unusual operations.

Figure 1 shows a flow-sheet for a diethylene glycol gas dehydration plant installed on a pipe line near Otis, Kansas.

The solution contactor (1) is in a gas pipe line, hence no controls are desired for either the pressure or rate of flow of the gas; these functions being determined by the line operation. The diagram shows an internal float control to hold the solution level constant in the base of the contactor. Our tendency for the past several years has been to use internal level controls in preference to the external or "kidney" type, for the following reasons: (a) They are not subject to freezing in cold weather; (b) With modern materials and workmanship, failures of the float practically never occur; (c) They are usually cheaper than the kidney type.

The glycol solution leaving the contactor carries a small amount of gas in solution, which is largely removed in the vent tank (2), in which just enough pressure is carried to deliver the solution through the heat-exchanger into the still. This venting pressure control is accomplished by another internal float, which operates a valve on the vent gas. This is an application of general principle No. 2, discussed above; both a back-pressure regulator and a liquid-level controller might have been used on this tank.

The liquid level in the base of the still (3) is controlled by an external or kidney type float. The reason for the kidney float here is that the liquid in the base of the still is boiling violently all the time; the small connections to the float case damp out the surface disturbances and allow the float to ride smoothly, preventing excessive wear of the mechanism.

The use of a pressure-reducing valve (4) for controlling steam supply to the glycol still reboiler is an example of the application of general principle No. 3. The obvious instrument for this purpose is a temperature controller, such as might be used for a stabilizer reboiler. However, the heat load on the reboiler is practically constant, and the still-base temperature is not important so long as it is high enough; therefore, the simple and comparatively cheap scheme used controls the base temperature with sufficient accuracy.

The solution circulating rate is controlled by a standard make of pump-stroke governor, another application of principle No. 3. This is a very small plant; hence the fuel which might be saved by proportioning the solution rate to the gas flow is negligible; therefore the stroke governor is set to deliver the maximum required rate, and left alone. In a larger plant a ratio controller to proportion the solution rate to the gas flow might well be justified.

Figure 2 is the flow-sheet of the distillate extraction and gas-pressure maintenance plant of the Corpus Christi Corporation. The general scheme involves cooling the gas and distillate mixture which comes from

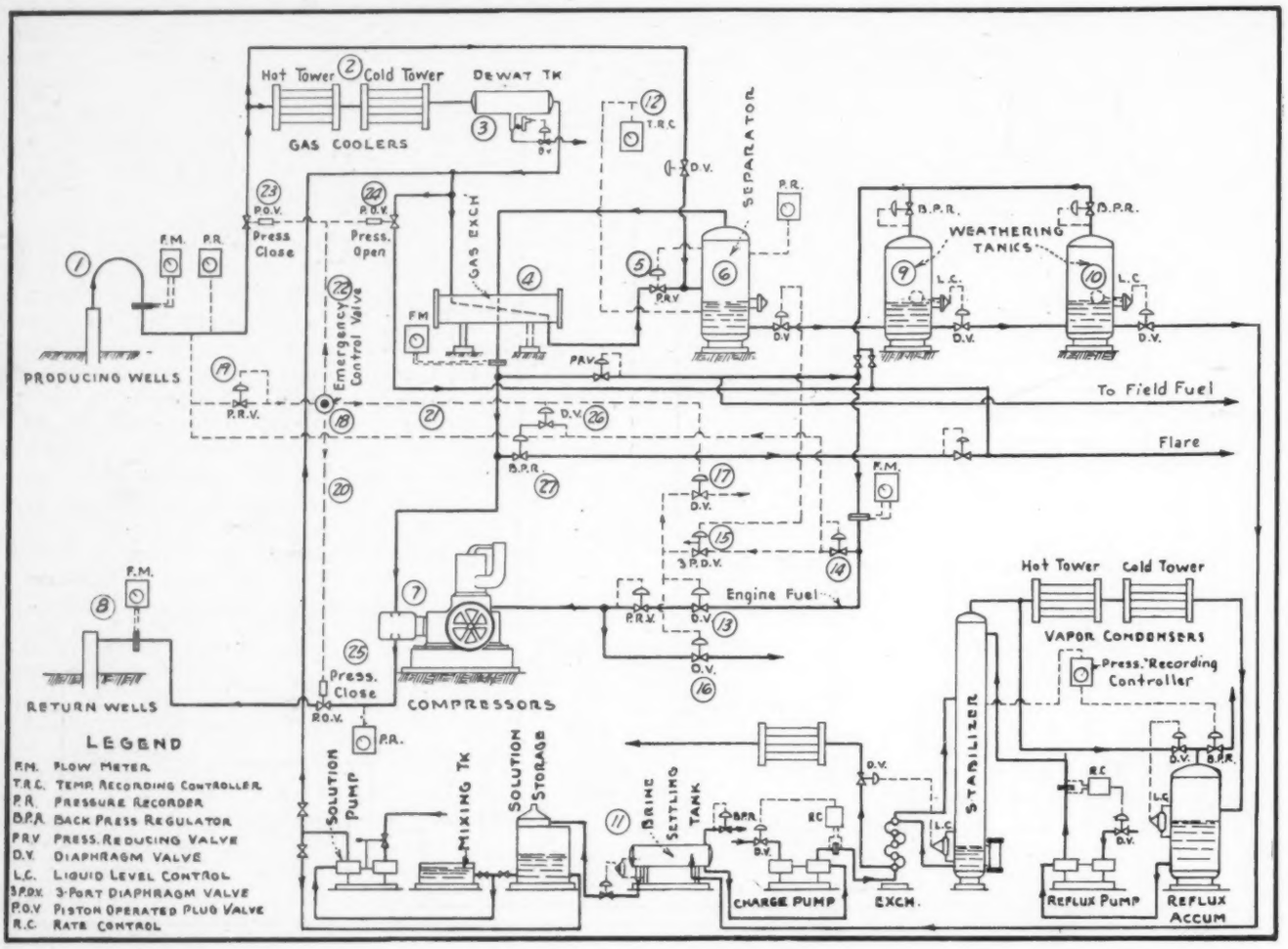


FIGURE 2

the wells, seperation of the distillate from the gas at as high a pressure as possible, return of the separated gas to the producing formation by compression, and stabilization of the separated distillate to 10-pound Reid vapor pressure. In order to prevent hydrate or freezing obstruction in the high-pressure part of the system, arrangements are provided to inject calcium chloride brine into the incoming gas. This brine is separated from the distillate just ahead of the stabilizer, is reconcentrated, and used continuously.

The main gas flow is from the producing wells (1), through atmospheric coolers (2), water separator (3), heat exchanger (4) and pressure-reducing valve (5) to separator (6). From the separator the gas passes back through the other side of the exchanger (4) directly to the suction of the pressure maintenance compressor (7), and thence to the return wells (8). A back-pressure regulator (27), normally responsive to the wet gas pressure at the plant inlet, serves to blow excess gas to the air in the event part or all of the compressors are shut down, until the necessary producing well adjustments can be made, restoring normal pressure conditions at the plant inlet.

The distillate flows from the separator through three stages of weathering under controlled pressures in tanks (9), (10) and (11); in the latter tank the brine is settled out and run off to the reconcentrating system. The stabilizer charge pump takes suction from this same tank. A temperature recorder-controller (12) can by-pass a small amount of hot gas around the coolers and exchanger, so as to maintain the exact desired temperature in the separator (6).

The elements of this process are very simple and conventional, but perhaps two of the safety provisions included warrant further discussion.

The first of these is the arrangement for protecting the compressors against liquid carry-over from the separator, in case a stoppage should occur in the distillate drain from it. The float in the separator operates a throttling type pilot, which delivers a variable pressure to its controlled valve according to the position of the float. The liquid control valve on the separator is designed to open wide when the pressure on its diaphragm reaches say 5 pounds per square inch, which corresponds to normal high level position of the float. If the level should continue to rise, the float pilot will deliver an increasing pressure into its controlled pilot line; this increase serves to shut down the compressors in the following manner:

Fuel gas to the engines is delivered through a spring-closed diaphragm valve (13), which is normally held open by pilot pressure delivered through regulator (14) and emergency level valve (15). This same pressure also holds closed bleeder valve (16), which is a spring-opened single-seated valve of large capacity, arranged to quickly bleed off pressure from the engine fuel header when pressure on its diaphragm is released. Emergency valve (15) is a 3-way valve, with its diaphragm connected to the separator float pilot. Normally it simply passes pilot pressure to the diaphragms of valves (13) and (16), holding them open and closed respectively, and thus permitting the engines to run. If the level in the separator should rise dangerously high, the float pilot will deliver a pressure to the diaphragm of 3-way valve (15) sufficiently high to cause it to operate, shutting off the supply of pilot gas from valve (14) and at the same time releasing pressure from the diaphragms of valves (13) and (16). This shuts off the supply of engine fuel, and also instantly blows down the pressure in the engine fuel header, stopping the machines. Valve (17) is normally closed, but can shut down the engines in the same manner, as will be discussed.

The other unusual safety arrangement in the plant is the provision of three emergency shut-down stations, from any one of which an operator, by simply turning a lever, can shut off all gas supply into the plant, blow down the pressure in the plant system, and shut down the compressors. It was felt that such arrangements were desirable on account of the high pressures and large volumes being handled; the volume averaging around 50 m.c.f. a day, and the pressures ranging up to 1800 pounds on the suction and 3000 pounds on the discharge of the compressors.

The manual emergency shut-down can be operated from any one of three stations, one of which is located in front of the plant office, another in the yard near the auxiliary building, and the third on the instrument panel in the compressor house. Each control station (one of which is indicated at (18) on the diagram) is simply a 1-inch lubricated plug cock with a handle permanently attached. A supply of gas at about 100 pounds a square inch pressure is delivered to the emergency valve through regulator (19). Opening an emergency valve therefore applies pressure simultaneously to lines (20), (21) and (22). Pressure is kept from building up in these lines from leakage through the emergency valves by providing small permanent bleeds, whose capacity is negligible as compared to the plug-cock capacity, and yet is sufficient to bleed off any leakage.

The application of pressure to line (22) simultaneously operates piston-operated plug valves (23) and (24), shutting off outside the plant fence the flow of wet gas into the plant, and opening a 3-inch valve to the stack line to blow down pressure from the system ahead of the separator. Applying pressure to line (20) closes a similar plug cock (25), which shuts off the gas return pipe system and thus prevents possible back-flow into a failure within the plant.

Pressure applied to line (21) opens diaphragm valve (17), releasing pressure from the pilot system which operates valves (13) and (16), thus shutting down the compressors as already described. The pressure also operates pilot diaphragm valve (26), which applies pressure to the diaphragm of main compressor suction relief regulator (27), causing the latter to open and blow down plant pressure to the stack line through a second channel.

Thus the operation of any one of these emergency valves almost instantly isolates the plant from the gathering and return pipe systems, shuts down the compressors, and blows down pressure within the plant through two 3-inch valves in parallel. The purpose of the system is of course to protect the plant from destruction which might result from a minor initial accident.

This last discussion illustrates the application of control equipment to promote plant safety, which is a function entirely distinct from the usual processing applications. Often, as in several of the details just described, the two functions can be combined by the addition of a small amount of auxiliary equipment, thus giving the plant important protection at small expense.

Progress in West Coast Manufacturing Activities

CALIFORNIA refinery, natural gasoline and liquefied petroleum gas operations continue at satisfactory levels even though the output of crude oil has not been reduced, and general business conditions are lower than at the close of 1938. Average daily crude oil production for May is estimated at 625,000 barrels.

No large-scale refinery construction programs are anticipated in the immediate future, although several remodernization jobs are planned. Gasoline-plant construction is at a standstill, but several field developments may soon attract attention in this direction.

FUEL OIL DISTRIBUTION

A preliminary estimate of the distribution of fuel oil on the Pacific Coast Territory (California, Oregon, Washington, Arizona and Nevada) during 1938 would be about 104,000,000 barrels. This estimated volume is nearly 500,000 barrels greater than the 102,578,000 barrels distributed during 1937. The volume of fuel oil used by railroads during 1938 is expected to be close to 28,000,000 barrels, or a 1,250,000-barrel decrease over the 29,276,000-barrel figure for 1937. During 1939 the fuel oil demand by railroads is expected to increase the 1938 figure because of augmented traffic schedules due to the San Francisco fair.

The chief users of fuel oils in descending order of demand are: railroads, marine vessels, smelters and manufacturing industries, general heating requirements, miscellaneous uses (trucks, tractors, etc.), U. S. Army, Navy and Coast Guard, and public utilities.

Data covering Pacific Coast exports of kerosene is still fragmentary. The Chinese market has been disorganized due to the war and orders for American kerosene during 1938 were sporadic. The volume of kerosene sold on the Pacific Coast during 1938 will probably be in the neighborhood of 1,700,000 barrels.

RESEARCH AND DEVELOPMENT

First effect of the trend toward catalytic cracking on the Pacific Coast was the recent announcement that Standard Oil Company of California had purchased a license to operate the Houdry catalytic process. Soon after came the report that Shell Oil Company, Richfield Oil Corporation and The Texas Company are also planning to build Universal catalytic conversion units. While it is not likely that The Texas Company will build a unit on the West Coast, Shell Oil Company and Richfield Oil Corporation are expected to be under way by the first part of 1940.

Opinions differ as to what constitutes a sound economic view of the possibilities of catalytic cracking in California. One view is that with good antiknock characteristics already available, catalytic processing is simply adding to refining costs to gain a few octane units. Another view is that catalytic processing is worth the cost and will pay off well through the reduction in the amount of lead used in first-grade and premium fuels. One medium-sized refinery in Southern California is known to pay over $400,000 a year for lead alone.

Some contend that the nature of the crude supply will be a factor in deciding whether to process catalytically or not. Companies which have substantial holdings of their own can curtail to suit supply and demand. On the other hand, many refiners are dependent upon large amounts of independently produced crude which is contracted for.

LABORATORY CONSTRUCTION

General Petroleum Corporation has begun construction of a new laboratory at its Torrance refinery. The cost is expected to be close to $20,000. With the old Vernon refinery no longer operating, Torrance has been growing rapidly. Research and development work is still done at Vernon and it is not yet known whether this work will also be housed in the new Torrance laboratory.

SYNTHETIC CHEMICALS FROM REFINERY GASES

An outstanding achievement on the Pacific Coast has been the development of manufacturing processes by Shell Oil Company for the synthesis of commercially important organic chemicals from selected refinery gases. Starting with propylene and the butylenes, various alcohols and other solvents known chemically as "ketones" are synthesized. The system used has been described as the preparation of pure hydrocarbon fractions containing only the desired "unsaturate" (propylene or butylene) and its corresponding "paraffin" (propane or butane). The unsaturate is absorbed in concentrated sulphuric acid, with which it is reactive, to produce what might be termed an organic sulphate. The organic sulphates are then "hydrolyzed" (water attached chemically) to form the desired alcohol (propyl or butyl). Substances known as "ethers" are formed with the alcohols. These are separated from the alcohol by distillation. The alcohols are further purified by fractionation.

From the alcohols (known as "secondary" alcohols) other products are made. If a portion of the hydrogen from an alcohol is removed a "ketone" results. If acetic acid is reacted with these "secondary" alcohols, substances known as "esters" result.

By other organic processes, powerful solvents known as diacetone alcohol, mesityl oxide and methyl isobutyl ketone result.

All of these solvents have wide commercial applications among which can be mentioned their use in lacquers, plastics, safety glass, photographic films, inks, medicines, explosives, and solvent de-waxing.

REFINERY BOILER WATER TREATMENT

An important problem which has always baffled power engineers is how to reduce the soda compounds in boiler feed water without having to distill it. It is well known that the conventional chemical methods of softening, whether by zeolite or lime-and-soda, always result in the accumulation of soda compounds.

The latest development in how to remove soda compounds without distilling is by means of synthetic organic substances which behave like zeolites. These substances are often spoken of as organic exchange filters.

The first installation of such a water-treating process in the Pacific Coast oil industry has been made by Standard Oil Company of California at its Richmond refinery.

While the composition of the water treated at Richmond is not known, the process can be illustrated by describing how it would remove the calcium sulphate from a "gyp" water. The first filter consists of an "acidic exchanger." As the gyp water passes through, the exchanger replaces the calcium in the calcium sulphate with hydrogen, forming dilute sulphuric acid. The water containing the dilute sulphuric acid then passes through the next filter which is known as the "basic exchanger." Here the sulphate portion of the dilute sulphuric acid is replaced by what is known chemically as an alkaline or "hydroxyl" group. After this action has taken place the gyp is completely removed. The calcium portion was left in the acidic filter, and the sulphate portion in the basic filter.

NATURAL GASOLINE OPERATIONS

While no new gasoline plants are being built at present, Pacific Coast manufacturers are carrying on repair and modernization work. With the end of the rainy season at hand, many are planning boiler stack and equipment painting jobs, cooling tower and heat exchanger cleanouts, and general "spring cleaning." Gasoline production is still at a high level and the volume for May will be close to 55,500,000 gallons.

ENGINEERING

At a recent meeting of the California Natural Gasoline Association, F. T. Patton discussed the question of recognizing and repairing weaknesses in pressure vessels. The talk stressed the need for particular attention to major weaknesses inherent in seams, heads and openings.

Some of the factors contributing to faulty fabrication were cited as (1) poor head shapes; (2) improperly reinforced openings in shells and heads; (3) improperly attached nozzles and reinforcing; (4) improperly located connections; (5) improperly designed or improperly located attachments, such as supporting brackets; (6) unconventional features of construction, such as large eccentric openings in heads.

Service factors which can weaken pressure vessels were summarized as: (1) corrosion; (2) embrittlement; (3) fatigue cracks from repeated reversals of stress; and oxidation or deformation by direct-firing.

In preparing to make an inspection of a pressure vessel it was pointed out that important elements to keep in mind are: (1) date of construction; (2) what code built to conform to; (3) initial and subsequent test pressures; (4) examination of original drawings to determine type of construction; (5) records of past inspections; and (6) service conditions.

In making the inspection the following were described as essential items to be determined: (1) thicknesses of shells and heads, by drilling and gauging if necessary; (2) check of riveted joints and seams; (3) visual inspection of inside and outside of welded seams; (4) check of shape of head; (5) check of head-to-shell attachment; (6) details of all connections to shell and heads; and (7) evidence of corrosion.

WATER TREATMENT

Economies possible through the use of hard water in cooling towers instead of the more costly soft waters has long attracted the attention of gasoline-plant engineers. Various schemes of using hard water have been tried with varying results.

A recent innovation to attract attention in this direction is the application of a chemical known as hexametaphosphate of soda to hard cooling-tower waters. Opinions differ as to what takes place, but one reliable source states that this chemical forms a complex combination with the calcium and magnesium hardness in the water, holding it in solution at temperatures up to 160-180° F. and thereby preventing the decomposition of the hardness and its accumulation as scale.

The most interesting part about the application of this chemical is that only small amounts are needed. From 2 to 5 parts per million has been recommended as sufficing for the most cases.

LIQUEFIED PETROLEUM GAS

The first quarter of 1939 finds the Pacific Coast liquefied petroleum gas industry actively expanding. The total production from all sources during May is expected to be close to 4,250,000 gallons. The use of butane for municipal fuel, and for fuel in large construction jobs continues to illustrate the gradual but sure increase in distribution outlets.

The most recent large scale use of liquefied petroleum gas on a construction job is in connection with the building of the Shasta Dam on the Sacramento River, near Redding, California. Butane storage consists of two 10,000-gallon tanks. The butane is delivered in 6000-gallon truck-and-trailer units which are unloaded with a four-stage electrically-driven pump.

A 3000-cubic-foot-per-hour butane vaporizing unit is also provided to make gas for fuel around the camp. The liquefied petroleum gas is vaporized at a pressure of 12 inches of water and distributed through a 3-inch main with 1½-inch laterals.

At Palm City, California, construction work has begun on a 4000-gallon storage and bulk distribution station for the South Bay Butane Company. Several similar bulk distribution stations are operated by other concerns in the San Joaquin and Sacramento Valleys. Small tank trailers are filled at the storage plant and used to deliver liquefied petroleum gas to farmers, small manufacturing shops, and other locations where storage cylinders too large to be exchanged are used.

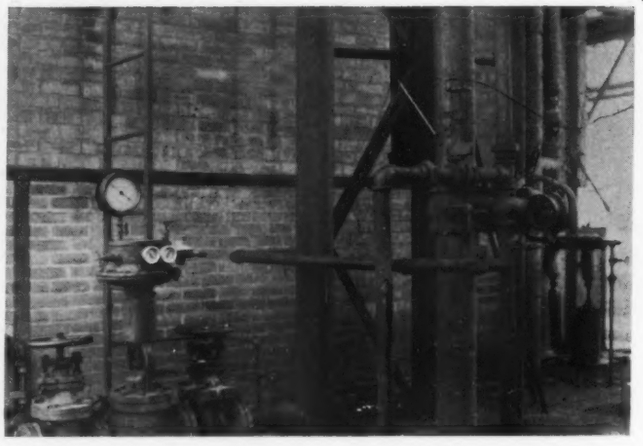
Engine Jackets *Used as* Auxiliary *Feedwater Heaters*

HAVING developed an unusually good supply of water for plant use, Talco Asphalt & Refining Company, Mt. Pleasant, Texas, found it unnecessary to treat either boiler feed or gas engine cooling water. The water used in this plant is produced at sufficient depth that it has a fairly constant year-round temperature, ideal for cooling, but not suitable for boiler-feed without preheating. The principal heating unit installed on the twin water-tube boilers is similar to that in service in a number of power plants, heating the feed-water to about atmospheric boiling temperature before the feed-water pumps take suction.

With an electric power generation plant in continuous operation, together with air compressors in operation the major portion of the day, a considerable volume of jacket water is required for cooling. The temperature of the outlet from jackets and exhaust coolers is much higher than the supply water to the plant, and to maintain the desired cooling water temperature, a cooling tower was required, together with hot wells and circulating pumps, or fresh water must flow through the jackets at all times, to either be lost or diverted to the plant storage reservoir.

As a large volume of steam is required for pipe line heating, where asphalt products are transferred from tank to tank, it is not feasible to recover condensate from distant points for reuse in the boilers. Consequently, an amount of make-up water comparable to jacket water needs is required for boilers, and to take advantage of the temperature of the outlet of the jackets and exhaust coolers, the water passes through the jackets once, and is then used for steaming. Lines were connected from deep-well pumps so the freshly produced water could flow under controlled volume through cooling units, jackets and the like, with a back-pressure suitable for make-up purposes and to pass through the regular boiler-feed heater.

This required a closed system on the engines designed to operate at approximately 30 pounds, with control valves adjusted to pass a volume of water so that adequate temperatures could be carried on the jackets and other parts of the engines and air compressors. Lines lead from the various units to the boilers, where an automatic by-pass is installed which allows jacket water to enter the feed-water heater automatically. When the boilers require as much or more than the volume passing through the engines, the water enters the heater, but if the volume exceeds the requirements, the automatic instrument by-passes excess fluid to drain lines leading to storage ponds and tankage provided for emergency water supply.



Piping and pressure regulator on boiler feedwater system that controls flow of water to heater unit from gas engines.

It is observed with this system that equipment cost has been reduced by the price of circulating pumps, hot well, spray pond and other accessories, and that the water entering the feed-water heater is high enough in intake temperature that heating is reduced to a minimum. The boiler-feed system and the cooling for gas engines, while connected to each other are in effect independent systems, either functioning without interference from the other. When the boilers do not require make-up water, the discharge from the cooling system is by-passed to ponds, and if the engines are down, the boilers take make-up from plant supply lines.

Where raw water is produced with a large total hardness, this system would not be practical, but if boilers required approximately the amount of make-up water that is necessary for jacket cooling, treated water might be passed first through jackets and then to the hot-well, directly to the preheater pump. The object of this hook-up is to reduce heating steam for feed-water for the boilers, which in turn reduces the load on the boilers themselves.

The only precaution necessary in this system is to provide an adequate automatic by-pass that functions positively, so that excessive pressure is not applied to the jackets of the units being cooled.